

SEP 12 1996

ENGINEERING DATA TRANSMITTAL

Page 1 of 1
1. EDT 617542

2. To: (Receiving Organization) Distribution	3. From: (Originating Organization) Data Assessment and Interpretation	4. Related EDT No.: N/A
5. Proj./Prog./Dept./Div.: Tank 241-C-204/Waste Management/DAI/TWRS Technical Basis	6. Design Authority/ Design Agent/Cog. Engr.: John M. Conner	7. Purchase Order No.: N/A
8. Originator Remarks: This document is being released into the supporting document system for retrievability purposes.		9. Equip./Component No.: N/A
		10. System/Bldg./Facility: 241-C-204
11. Receiver Remarks: 11A. Design Baseline Document? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No For release.		12. Major Assm. Dwg. No.: N/A
		13. Permit/Permit Application No.: N/A
		14. Required Response Date: 09/05/96

15. DATA TRANSMITTED					(F)	(G)	(H)	(I)
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1	WHC-SD-WM-ER-479	N/A	0	Tank Characterization Report for Single-Shell Tank 241-C-204	N/A	2	1	1
16. KEY								
Approval Designator (F)		Reason for Transmittal (G)			Disposition (H) & (I)			
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)		1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)			1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged			
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BD-7400-172-2 (05/96) GEF097

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Tank Characterization Report for Single-Shell Tank 241-C-204

John M. Conner
Westinghouse Hanford Company, Richland, WA 99352
U.S. Department of Energy Contract DE-AC06-87RL10930

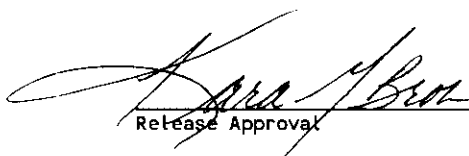
EDT/ECN: EDT-617542 UC: 2070
Org Code: 79400 Charge Code: N4G4D
B&R Code: EW 3120074 Total Pages: 78

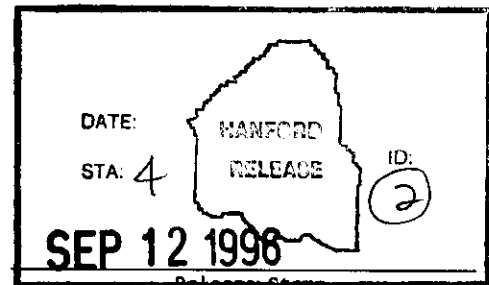
Key Words: Tank Characterization Report, TCR, Single-Shell Tank,
Single-Shell, SST, Tank 241-C-204, Tank C-204, C-204, C Farm

Abstract: This document summarizes the information on the historical
uses, present status, and the sampling and analysis results of waste
stored in Tank 241-C-204. This report supports the requirements of Tri-
Party Agreement Milestone M-44-09.

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Tank Characterization Report for Single-Shell Tank 241-C-204

J. M. Conner

Date Published
September 1996

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management



**Westinghouse
Hanford Company**

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Management and Operations Contractor for the
U.S. Department of Energy under Contract DE-AC06-87RL10930

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EXECUTIVE SUMMARY

This tank characterization report summarizes the information on the historical uses, present status, and the sampling and analysis results of waste stored in single-shell underground storage tank 241-C-204 at the Hanford Site. This report supports the requirements of *Hanford Federal Facility Agreement and Consent Order*, Milestone M-44-09 (Ecology et al. 1996). Analytical results indicate the tank may pose a safety concern based on the decision limits of the *Tank Safety Screening Data Quality Objective* (Dukelow et al. 1995). Although tank samples exhibit energetics potential and total organic carbon (TOC) in excess of the limits in the data quality objective (DQO), the tank contains sufficient moisture to mitigate an exothermic event. Adiabatic calorimetry results indicate the waste will not propagate a reaction. Therefore, no imminent safety concern exists. Because a rag was encountered directly under the sampling riser and sampling was not completely successful, resampling is recommended.

Tank 241-C-204 is one of 16 single-shell underground waste storage tanks located in the C Tank Farm in the 200 East Area of the Hanford Site. The maximum capacity of tank 241-C-204 is 210 kL (55 kgal). The tank was filled with waste in January, 1948. Supernatant liquor was pumped from the tank in 1953, and water was added. The metal waste was removed for uranium recovery in early 1955, and the tank began receiving strontium semiworks waste late that same year. The tank received transfers of waste from the Hot Semiworks Plant in May 1956 and in the fourth quarter of 1967. Supernate was pumped from the tank in 1970 and 1977 leaving an 11 kL (3 kgal) heel of solids. Because

the Hot Semiworks Plant was operated as a pilot plant for separations processes, it is difficult to estimate the contents of the tank based on process history. Estimates generated prior to sampling assumed the remaining tank waste was metal waste. Based on sampling results, it is believed that little metal waste remains in the tank.

A description and status of the tank are summarized in Table ES-1. The tank currently contains 11 kL (3 kgal) of waste in the form of sludge. The latest tank photographs (1986) show a mixed yellow and brown, wet surface with crust near the walls. When the 1988 photographs are compared with earlier ones (1977, 1980, and 1983), it is evident the tank contents are slowly drying out. Temperature data, available for 1975 to 1978, indicate the tank temperature remained below 21 °C (70 °F) during this period. Although thermocouple elevations are not known, it is assumed that thermocouples were in the waste prior to tank liquids being pumped in 1977. The current waste level is approximately 0.4 m (1.3 ft).

This report describes data from the May 1995 auger sampling event. Auger samples 95-AUG-022 and 95-AUG-023 were taken and analyzed according to the requirements of the safety screening DQO. Energetics, moisture, and total alpha content were determined. Secondary analyses were performed to determine the TOC content and to test for energetic potential under adiabatic conditions. In addition, one sample was analyzed for organic compounds at the request of the Organic Safety Program. Data from the June 1996 tank headspace flammability screening are reported as well.

Table ES-1. Description and Status of Tank 241-C-204.

TANK DESCRIPTION	
Type	Single-shell
Constructed	1943 to 1944
In service	1948 to 1977
Diameter	6.1 m (20 ft)
Usable depth	5.2 m (17 ft)
Operating capacity	208 kL (55 kgal)
Bottom shape	Dish
Ventilation	Passive (breather filter)
TANK STATUS	
Total waste volume (February 1996)	11 kL (3 kgal) ¹
Sludge volume	11 kL (3 kgal) ¹
Saltcake volume	none
Supernatant volume	none
Surface level (manual tape)	0.4 m (1.3 ft)
Temperature (1975 to 1977) ²	13 - 20 °C (55 - 68 °F)
Integrity category	Assumed leaker
Watch List status	None at present
SAMPLING DATES	
Two auger samples	May 1995
Flammability screening	June 1996
Vapor sampling	July 1996 ³
SERVICE STATUS	
Inactive	1978
Interim stabilized	1982
Intrusion prevention	1982

Notes:

¹Hanlon (1996)²No temperature data is available after 1977.³No data is available at this time.

Because of limited riser availability, both auger samples were taken from the same riser. Unfortunately, the augers encountered a rag that was apparently dropped on the surface. In retrospect, it is apparent the auger samples did not retrieve a full-length profile of tank waste. Nevertheless, sufficient tank waste material was segregated to complete the analyses prescribed by the safety screening DQO. The results of the auger sample analyses indicate the tank waste has an unexpectedly high energetics potential, well in excess of the 481 J/g action limit. For most samples, a final value for the exotherm could not be obtained as the exotherm was still progressing at the temperature limit of the test. Results in excess of 1,234 J/g were reported. Secondary analyses were conducted to measure the TOC content of the samples. The TOC concentration was 13 weight percent (26 percent on a dry basis). Organic speciation showed that the organic component of the waste is almost exclusively tributyl phosphate, which was used as an organic solvent in several separations processes at the Hanford Site. Total inorganic carbon (TIC) was determined incidentally. The waste has a moisture content of approximately 55 percent. This is sufficient to preclude the possibility of an energetic reaction in the tank at this time. The adiabatic calorimetry results also indicate the waste will not propagate a reaction. The total alpha levels of the 1995 auger samples averaged 0.0266 $\mu\text{Ci/g}$. These levels are far below the safety screening limit of 41 $\mu\text{Ci/g}$, and they indicate no criticality concerns exist. A summary of results from the 1995 auger samples is shown in Table ES-2.

Table ES-2. Chemical Summary for Tank 241-C-204.

Analyte	Overall Mean Concentration	Relative Standard Deviation	Projected Inventory ¹
RADIONUCLIDES	$\mu\text{Ci/g}$	%	Cl
Total alpha	0.0322	60.5	0.602
CARBON	$\mu\text{g C/g}$	%	kg
TIC	10,500	16.9	196
TOC	126,000 (WHC) 60,000 ² (PNNL)	10.3 Not reported	2,360 1,120
ORGANICS²	$\mu\text{g/g}$		kg
Tributyl phosphate	330,000 ²	Not reported	1,190
Dibutyl phosphate	2,000 ²	Not reported	37
Monobutyl phosphate	None ²	Not reported	None
Chelators, formate, acetate, oxalate, normal paraffin hydrocarbons, butyric acid, toluene, benzoic acid	Trace ²	Not reported	Trace
PHYSICAL PROPERTIES	%	%	kg
Percent Water	56.95	2.15	10,600
THERMODYNAMIC PROPERTIES	Joules/gram (dry basis)		
Energetics by DSC	Range 813 to > 1,234	n/a	n/a
FLAMMABLE GAS	PERCENT OF LFL IN HEADSPACE		
Combustible gas meter screening	0	n/a	n/a

Notes:

WHC = Westinghouse Hanford Company
PNNL = Pacific Northwest National Laboratory

¹Based on an estimated mass of 18,700 kg (Brevick et al. 1994).

²These analyses were not conducted to the quality assurance requirements of the SAP and should be used with caution.

The tank headspace was screened with a combustible gas meter. The results indicated that the headspace was at 0 percent of the LFL. Headspace gas sampling has been conducted, but analysis is not complete. Results will be included in a revision of this document.

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LIST OF TERMS

Btu/hr	British thermal units per hour
Ci	curies
Ci/L	curies per liter
cm	centimeters
cm ³	cubic centimeters
DQO	data quality objective
DSC	differential scanning calorimetry
FIC	Food Instrument Corporation
ft	feet
g	gram
HDW	Hanford Defined Waste
in.	inches
J/g	joules per gram
kg	kilograms
kgal	kilogallons
kL	kiloliters
kW	kilowatt
L	liters
LFL	lower flammability limit
mole/L	moles per liter
m	meters
mR/hr	milliroentgen/hour
ppm	parts per million
RPD	relative percent difference
RSD	relative standard deviation
SAP	sampling and analysis plan
TGA	thermogravimetric analysis
TIC	total inorganic carbon
TLM	tank layer model
TOC	total organic carbon
TWRS	Tank Waste Remediation System
wt%	weight percent
°C	degrees Celsius
°F	degrees Fahrenheit
μCi/g	microcuries per gram
μg C/g	micrograms of carbon per gram
μg/g	micrograms per gram
ΔH	change in enthalpy

1.0 INTRODUCTION

This tank characterization report summarizes the information on the historical uses, current status, and sampling and analysis results of waste stored in single-shell tank 241-C-204. The tank was sampled in 1995 to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). Flammability screening was later performed to satisfy a subsequent revision of this data quality objective (DQO) (Dukelow et al. 1995). Vapor sampling and analysis is being performed to satisfy the *Data Quality Objective for Tank Hazardous Vapor Safety Screening* (Osborne and Buckley 1995). Analysis of vapor samples has not been completed. Results will be included in a revision of this report.

Tank 241-C-204 has been removed from service and interim stabilized. No further waste transfers are anticipated until the remaining tank waste is retrieved. (Current waste management strategy is to retrieve all tank wastes, pretreat to separate them into low- and high-level waste streams, and vitrify each stream prior to disposal.) The tank is not currently on any Watch List although the results of auger sampling indicate the tank contains a very high concentration of organic solvent (tributyl phosphate).

1.1 PURPOSE

The purpose of this report is to summarize information about the use and contents of tank 241-C-204. When possible, this information will be used to assess issues associated with safety, operations, environmental, and process development activities. This report also provides a reference point for obtaining more detailed information about tank 241-C-204.

1.2 SCOPE

This report describes the design, configuration, and waste history of the tank. It provides estimates of the chemical and radiochemical inventory of the tank from models based on tank transfer history and from waste streams assumed in the tank. These estimates have not been validated. The discussion of sampling data focuses on auger samples taken in 1995. Finally, the report discusses the results of the June 1996 flammability screening of the tank headspace.

The auger samples taken in 1995 were intended to support screening of tank 241-C-204 for potential safety issues. Test results were evaluated against the criteria in the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994 and Dukelow et al. 1995). Primary analyses included the following: differential scanning calorimetry (DSC) to evaluate fuel level and energetics potential, thermogravimetric analysis (TGA) to determine moisture content, and total alpha activity to evaluate criticality potential. High DSC results prompted secondary analyses that included TOC and adiabatic calorimetry by the Reactive System

Screening Tool. The TIC was also determined. Organic speciation was conducted to determine major organic waste constituents. Because organic speciation was not conducted to the quality assurance protocol called for in Conner (1996a), the data should be used with caution.

The tank headspace was screened for flammability concerns with a combustible gas meter to comply with the safety screening DQO (Dukelow et al. 1995).

2.0 HISTORICAL TANK INFORMATION

This section describes tank 241-C-204 based on historical information. It includes information on the current condition of the tank, tank design, transfer history, the process sources that have contributed to tank waste, and provides an estimate of the current contents based on the process history. It also describes events that may be related to tank safety issues, such as potentially hazardous tank contents or off-normal operating temperatures, and it summarizes available surveillance data. Solid and liquid level data are used to determine tank integrity (leaks) and to provide clues to internal activity in the solid layers of the tank. Temperature data are provided to evaluate the heat generating characteristics of the waste.

2.1 TANK STATUS

As of February 29, 1996, tank 241-C-204 contained an estimated 11 kL (3 kgal) of noncomplexed waste. The liquid waste volume was determined by a photographic evaluation. The solid waste volume was last updated using a manual tape surface gauge reading and photographs on April 28, 1982. The waste phases in the tank are shown in Table 2-1.

Table 2-1. Estimated Tank Contents.¹

Waste Form	Estimated Volume	
	Kiloliters	(Kilogallons)
Total waste	11	(3)
Supernatant liquid	0	(0)
Drainable interstitial liquid	0	(0)
Drainable liquid remaining	0	(0)
Pumpable liquid remaining	0	(0)
Sludge	11	(3)
Saltcake	0	(0)

Note:

¹Hanlon (1996)

The tank is a low-heat load tank that is passively ventilated and categorized as an assumed leaker. Interim stabilization and intrusion prevention are completed. Tank 241-C-204 is not on any Watch Lists. All monitoring systems were in compliance with documented standards as of February 29, 1996, except for temperature readings. (The thermocouple tree was out of service) (Hanlon 1996).

2.2 TANK DESIGN AND BACKGROUND

The 241-C Tank Farm is a first generation tank farm that was constructed in 1943 and 1944. The 241-C Tank Farm contains 12 100-series 2,006 kL (530 kgal) tanks and four 200-series 208 kL (55 kgal) tanks. The 200-series tanks have a diameter of 6 m (20 ft) and an operating depth of 7.5 m (24.5 ft). The tanks were designed for nonboiling waste with a maximum fluid temperature of 104 °C (220 °F) (Brevick et al. 1994a). Equipment to monitor and maintain the waste is sparse.

Tank 241-C-204 entered service in January 1948. The single-shell tank is constructed of 30-cm (1-ft)-thick reinforced concrete with a 6.4 mm (1/4 in.) mild carbon steel liner (ASTM A283 Grade C) on the bottom and sides (to within 15.2 cm [6 in.] from the top) and a 30-cm (1-ft)-thick flat concrete top. These tanks have a 15 cm (6 in.) dished bottom with a 90 cm (3 ft) radius knuckle. The bottom center elevation of tank 241-C-204 is 185 m (608.5 ft). The four smaller tanks of the C Tank Farm are not connected with cascade lines, but they are connected to a common diversion box. The tank is set on a reinforced concrete foundation. Four coats of primer paint were sprayed on all exposed interior tank surfaces. Tank ceiling domes were covered with three applications of magnesium zincfluorosilicate wash. Lead flashing was used to protect the joint where the steel liner met the concrete dome. Asbestos gaskets were used to seal the manholes in the tank dome. The tank was waterproofed on the sides and top with tar and gunite. The tank was covered with approximately 3.5 m (11.5 ft) of overburden.

Table 2-2 lists the tank risers. Figure 2-1 shows a plan view of the riser configuration and location. Tank 241-C-204 has a construction manhole and six grade-level risers. Interior tank photographs show four additional risers that are not accessible at grade level but which could be used with considerable effort. Figure 2-2 shows a tank cross section showing the approximate waste level and a schematic of the tank equipment.

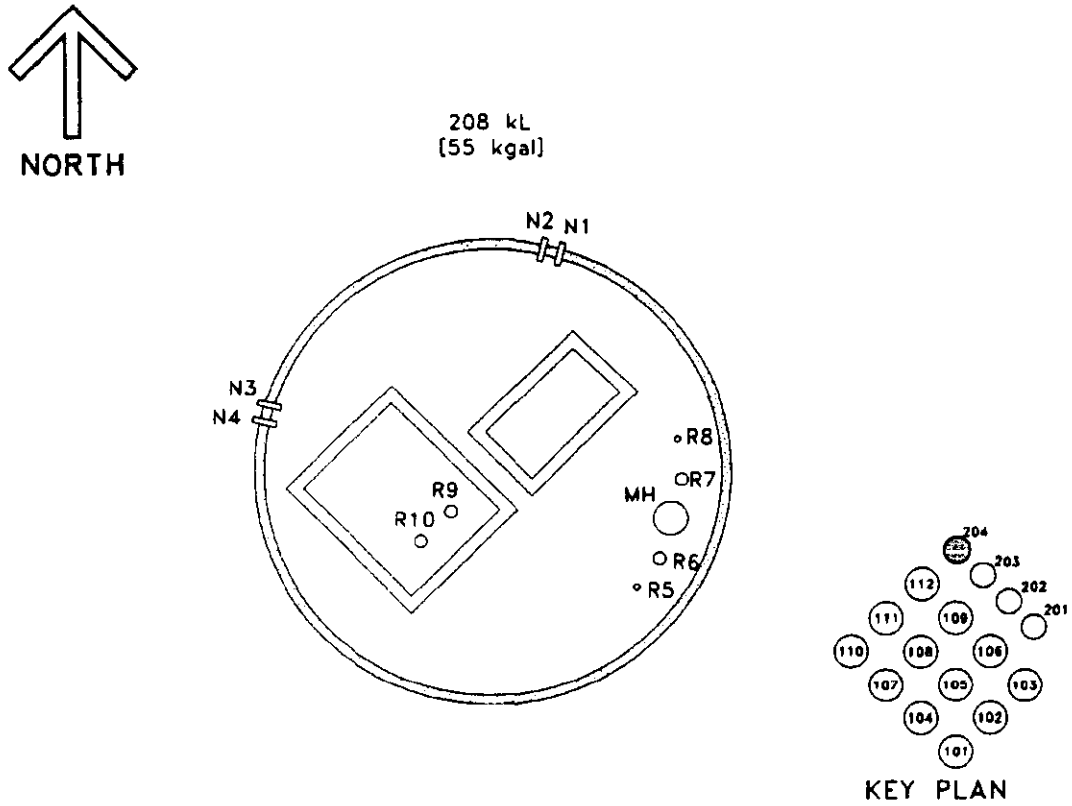
Table 2-2. Tank 241-C-204 Risers.^{1, 2, 3}

Riser Number	Diameter (In.)	Description and Comments
5	4	Liquid level reel
6	12	Unknown (thermocouple tree prior to 1977)
7	12	Flange/observation port
8	4	Breather filter
9	12	Sludge jet access, weather covered (pit)
10	12	Sluicing access, weather covered (pit)
Nozzle Number	Diameter (In.)	Description and Comments
1	3	Spare
2	3	Spare
3	3	Inlet
4	3	Inlet

Notes:

¹Alstad (1993)²Tran (1993)³Vitro Engineering Corporation (1986)

Figure 2-1. Riser Configuration for Tank 241-C-204.



2.3 PROCESS KNOWLEDGE

This section describes the transfer history of tank 241-C-204 and estimates current tank contents. Table 2-3 summarizes the waste transfers.

2.3.1 Waste Transfer History

In January 1948, tank 241-C-204 began receiving B Plant metal waste from tank 241-C-203; it was declared full by the end of the month. During the second quarter of 1953, its entire contents of supernatant metal waste was sent to tank 241-C-106, and flush water additions refilled the tank. The flush water was noted as containing metal waste (Agnew et al. 1995b). In the fourth quarter of 1953, waste from tank 241-C-204 was sent to uranium recovery operations in building 221-U (U-Plant). In the first quarter of 1954, additional flush water containing metal waste was received by the tank.

During the fourth quarter of 1954, supernatant metal waste was sent to uranium recovery operations in the building 221-U, and additional supernatant metal waste was received from tank 241-C-201. The receiving and sending of metal waste left tank 241-C-204 almost full. In the first quarter of 1955, the tank was emptied with a transfer of the liquid contents to uranium recovery operations in building 221-U.

The tank remained empty until the fourth quarter of 1955 when waste from the Hot Semiworks Plant was added. During the second quarter of 1956, additional hot semiworks waste was received.

Although the level history (Figure 2-4) shows a spike in 1957, the tank remained static, and no transfers are recorded until the addition of waste from the Hot Semiworks Plant in the third quarter of 1967. The waste was from strontium recovery operations. In the second quarter of 1970, supernatant waste was transferred to tank 241-C-104.

During the third quarter of 1977, 155 kL (41 kgal) was transferred to an unknown receiver. In 1977, the tank was declared inactive. In September 1982, the tank was administratively interim stabilized; in December 1982, intrusion prevention was completed. It was categorized as an assumed leaker in 1988 with a leak volume of 1,300 L (350 gal). Currently, the tank waste is classified as noncomplexed.

2.3.2 Historical Estimation of Tank Contents

The following estimate of the contents for tank 241-C-204 is based on historical transfer data. The data used in the estimate are from the *Waste Status and Transaction Record Summary for the Northeast Quadrant* (Agnew et al. 1995b), the *Hanford Tank Chemical and*

Table 2-3. Summary of Tank 241-C-204 Waste Transfer History.¹

Transfer Source	Destination	Waste Type ²	Time Period	Estimated Waste Volume ²	
				Kiloliters	(Kilogallons)
241-C-203	241-C-204	Supernatant metal waste	1948	208	(55)
241-C-204	241-C-106	Supernatant metal waste	1953	-208	(-55)
Unknown	241-C-204	Metal waste flush water	1953 to 1954	208	(55)
241-C-204	Uranium Recovery	Metal waste	1953	-150	(-40)
241-C-201	241-C-204	Supernatant metal waste	1954	200	(53)
241-C-204	Uranium Recovery	Metal waste	1954	-215	(-57)
241-C-201	241-C-204	Supernatant metal waste	1954	200	(53)
241-C-204	Uranium Recovery	Metal waste	1955	-200	(-53)
Hot Semiworks	241-C-204	Hot Semiworks waste	1955 to 1956	129	(34)
Hot Semiworks	241-C-204	Strontium recovery waste	1967	72	(19)
241-C-204	241-C-104	Supernate	1970	-53	(-14)
241-C-204	Unknown	Supernate	1977	-155	(-41)

Notes:

¹Agnew et al. (1995b)²Waste volumes and types are best estimates based on historical data. Some small transfers and level adjustments are not reported here. Refer to Figure 2-4 for a more accurate depiction of the level history.

Radionuclide Compositions: HDW Model Rev. 3 (Agnew et al. 1996), the *Tank Layer Model (TLM)* (Agnew et al. 1995a), and the *Historical Tank Content Estimate for the Northeast Quadrant of the Hanford 200 East Area* (Brevick et al. 1994a). The waste status and transaction record summary (WSTRS) is a compilation of available waste transfer and volume status data. The Hanford defined waste (HDW) list provides the assumed typical compositions for 50 separate waste types. In most cases, the available data are incomplete, thereby reducing the reliability of the transfer data and the derived modeling results. The TLM, using theWSTRS data, models the waste deposition processes and, using additional data from the HDW, generates an estimate of the tank contents. Several errors are introduced as the models are added to create the estimate; therefore, the model predictions

are estimates that require further evaluation using analytical data and should be used with caution. Agnew et al. (1996) states that tank 241-C-204 contains 11 kL (3 kgal) of waste, consisting of 4 kL (1 kgal) of hot semiworks waste and 7.5 kL (2 kgal) of metal waste. Figure 2-3 shows the estimated waste type and volume for the waste. The hot semiworks layer should contain large amounts of sodium, nitrate, and strontium with some acetate. The metal waste layer should contain large amounts of sodium, iron, hydroxide, carbonate, phosphate, nitrate, sulphate, and uranium. Chromium, calcium, nickel, nitrite, chloride, silicate, and a trace of plutonium will be found also. Small quantities of strontium and cesium will be present giving this layer a small activity. Table 2-4 shows an estimate of the expected waste constituents and their concentrations.

2.4 SURVEILLANCE DATA

Tank 241-C-204 surveillance consists of surface level measurements inside the tank. Tank 241-C-204 no longer has temperature monitoring equipment. Tank 241-C-204 has no liquid observation well or drywells.

2.4.1 Surface Level Readings

The tank 241-C-204 surface level is monitored quarterly with a manual tape through riser 5. Although the reported volume has not changed, the surface level dropped from 46 cm (18 in.) to 20.0 cm (8 in.) between December 1983 and January 1991. After fluctuating between 20 cm (8 in.) and 28 cm (11 in.) between January 1992 and May 1994, the surface level rose to 41 cm (16 in.) where it has remained. The last surface level reading was 41 cm (16 in.) on April 1, 1996. Figure 2-4 shows the tank level history from 1948.

Figure 2-5 shows the fluctuating level history, as measured by manual tape from 1981 to 1996. The drop in the measured level over time is probably caused by the sludge measurement weight creating a deeper and deeper hole when dropped to the surface of the tank. The recent increase to 41 cm (16 in.) might be caused by a collapse of material into the hole.

Figure 2-3. Tank Layer Model for Tank 241-C-204.

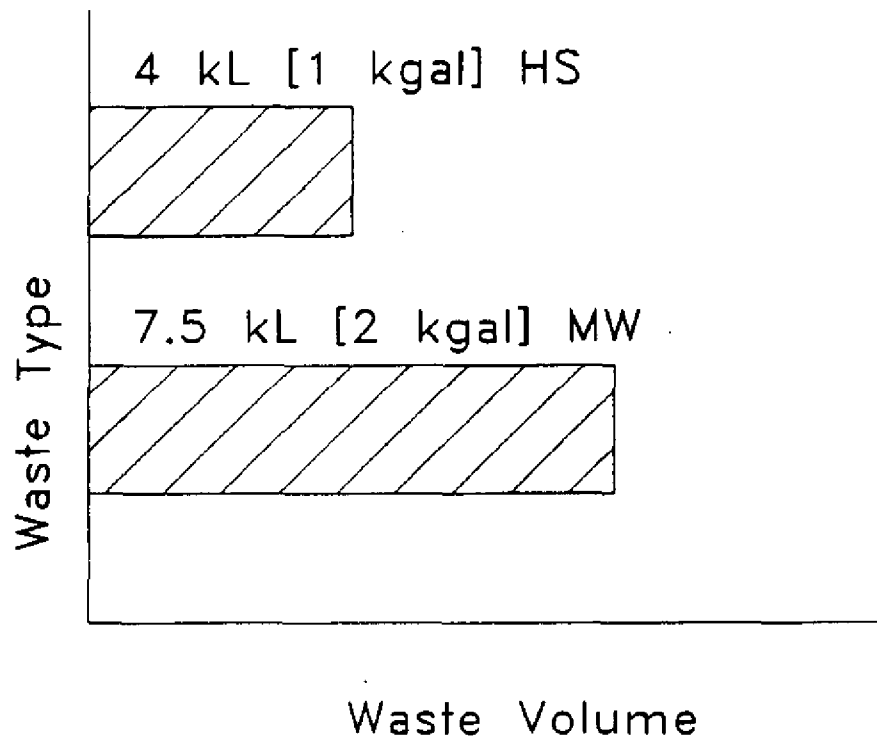


Table 2-4. Tank 241-C-204 Historical Inventory Estimate.^{1, 2} (2 sheets)

Physical Properties			
Total solid waste	1.87E+04 kg (3.00 kgal)		
Heat load	0.460 kW (1.57E+03 Btu/hr)		
Bulk density	1.65 g/cm ³		
Water	44.1 wt%		
TOC	0.412 wt% carbon (wet)		
Chemical Constituents	mole/L ³	ppm ³	kg ³
Na ⁺	3.97	5.54E+04	1.04E+03
Al ³⁺	0	0	0
Fe ³⁺ (total Fe)	1.97	6.67E+04	1.25E+03
Cr ³⁺	3.02E-03	95.5	1.78
Bi ³⁺	0	0	0
La ³⁺	0	0	0
Hg ²⁺	0	0	0
Zr (as ZrO(OH) ₂)	0	0	0
Pb ²⁺	5.05E-02	6.35E+03	119
Ni ²⁺	6.21E-02	2.21E+03	41.4
Sr ²⁺	0	0	0
Mn ⁴⁺	0	0	0
Ca ²⁺	5.74E-02	1.40E+03	26.1
K ¹⁺	2.45E-02	583	10.9
OH ⁻	14.1	1.46E+05	2.72E+03
NO ₃ ⁻	4.16E-02	1.57E+03	29.3
NO ₂ ⁻	0.305	8.52E+03	159
CO ₃ ²⁻	1.26	4.59E+04	858
PO ₄ ³⁻	0.267	1.54E+04	287

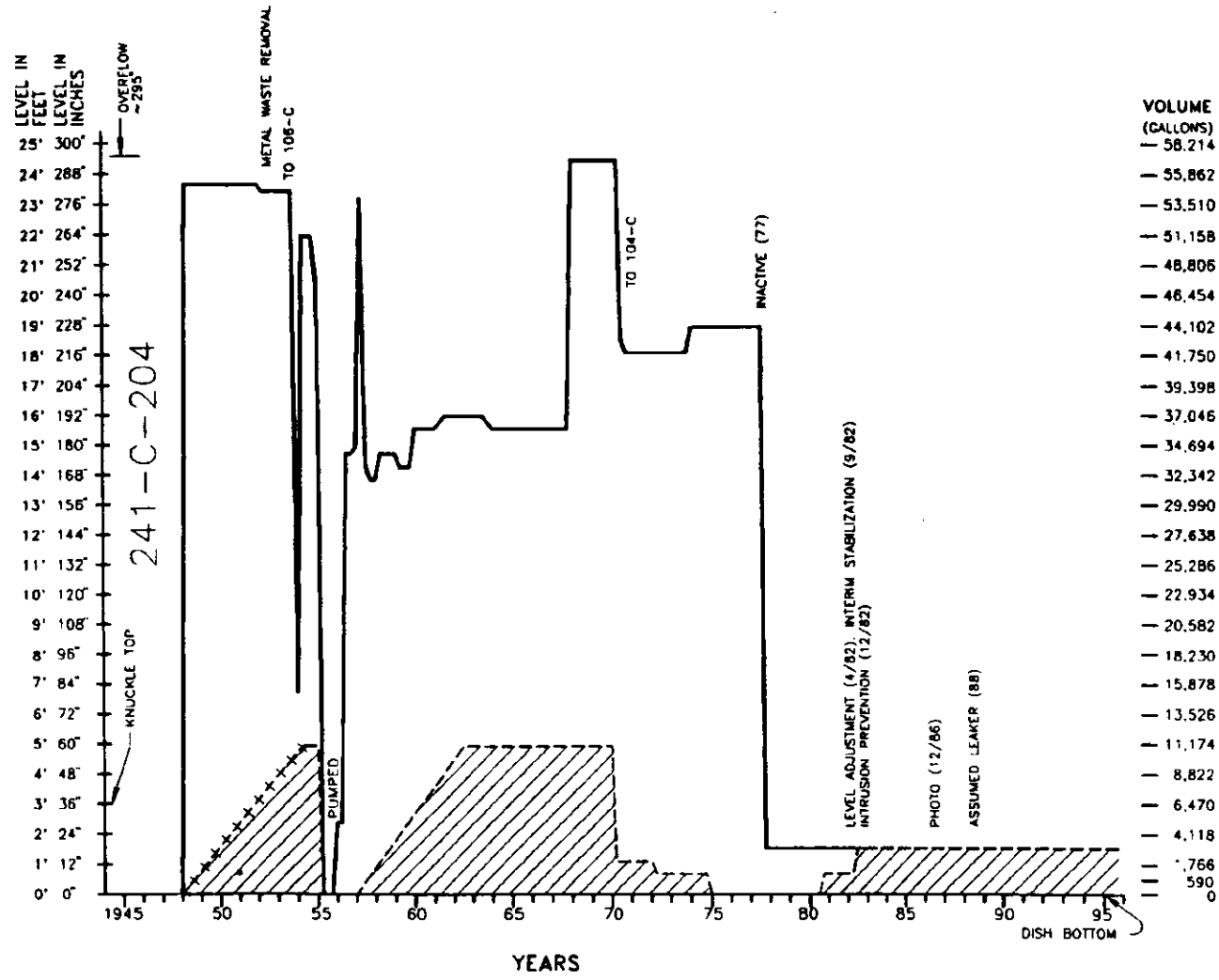
Table 2-4. Tank 241-C-204 Historical Inventory Estimate.^{1, 2} (2 sheets)

Chemical Constituents (Cont'd)	mole/L	ppm	kg
SO ₄ ²⁻	7.32E-02	4.27E+03	79.8
Si (as SiO ₃ ²⁻)	1.03E-03	17.6	0.330
F ⁻	0	0	0
Cl ⁻	1.43E-02	309	5.77
C ₆ H ₅ O ₇ ³⁻	1.10E-02	1.26E+03	23.6
EDTA ⁴⁻	2.20E-02	3.84E+03	71.8
HEDTA ³⁻	0	0	0
glycolate ⁻	0	0	0
acetate ⁻	0.140	5.02E+03	93.8
oxalate ²⁻	0	0	0
DBP	0	0	0
Butanol	0	0	0
NH ₃	0.148	1.53E+03	28.6
Fe(CN) ₆ ⁴⁻	0	0	0
Radiological Constituents			
Pu		2.63E-03 (μCi/g)	8.18E-04 (kg)
U	1.33 (M)	1.92E+05 (μg/g)	3.58E+03 (kg)
Cs	5.60E-04 (Ci/L)	0.340 (μCi/g)	6.36 (Ci)
Sr	6.01 (Ci/L)	3.65E+03 (μCi/g)	6.82E+04 (Ci)

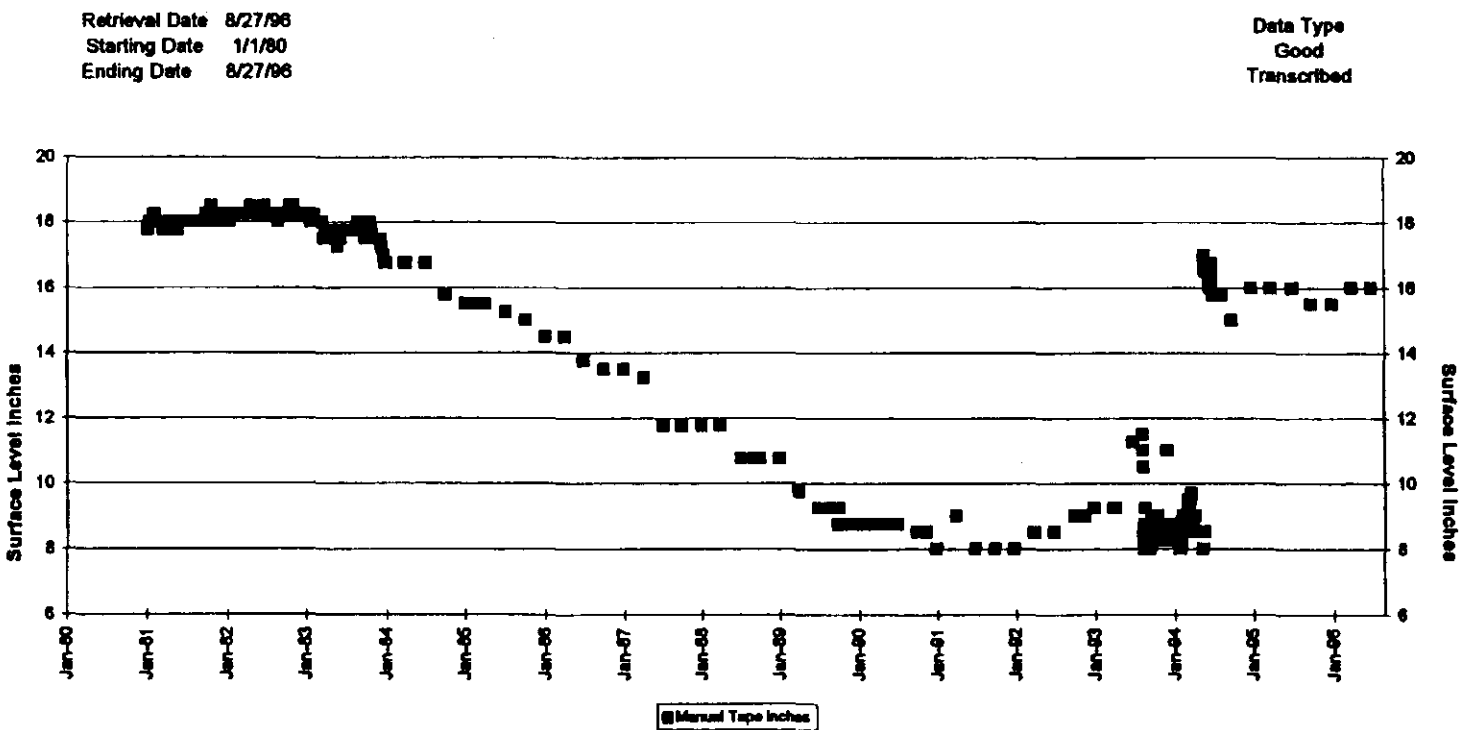
Notes:

¹Agnew et al. (1996)²The HTCE predictions have not been validated and should be used with caution.³When an inventory is calculated from the concentration values (using the density or waste volume), and those values are compared with the stated inventory in kilograms or curies, differences appear to exist. These differences are being evaluated.

Figure 2-4. Tank 241-C-204 Level History.



PH5E066



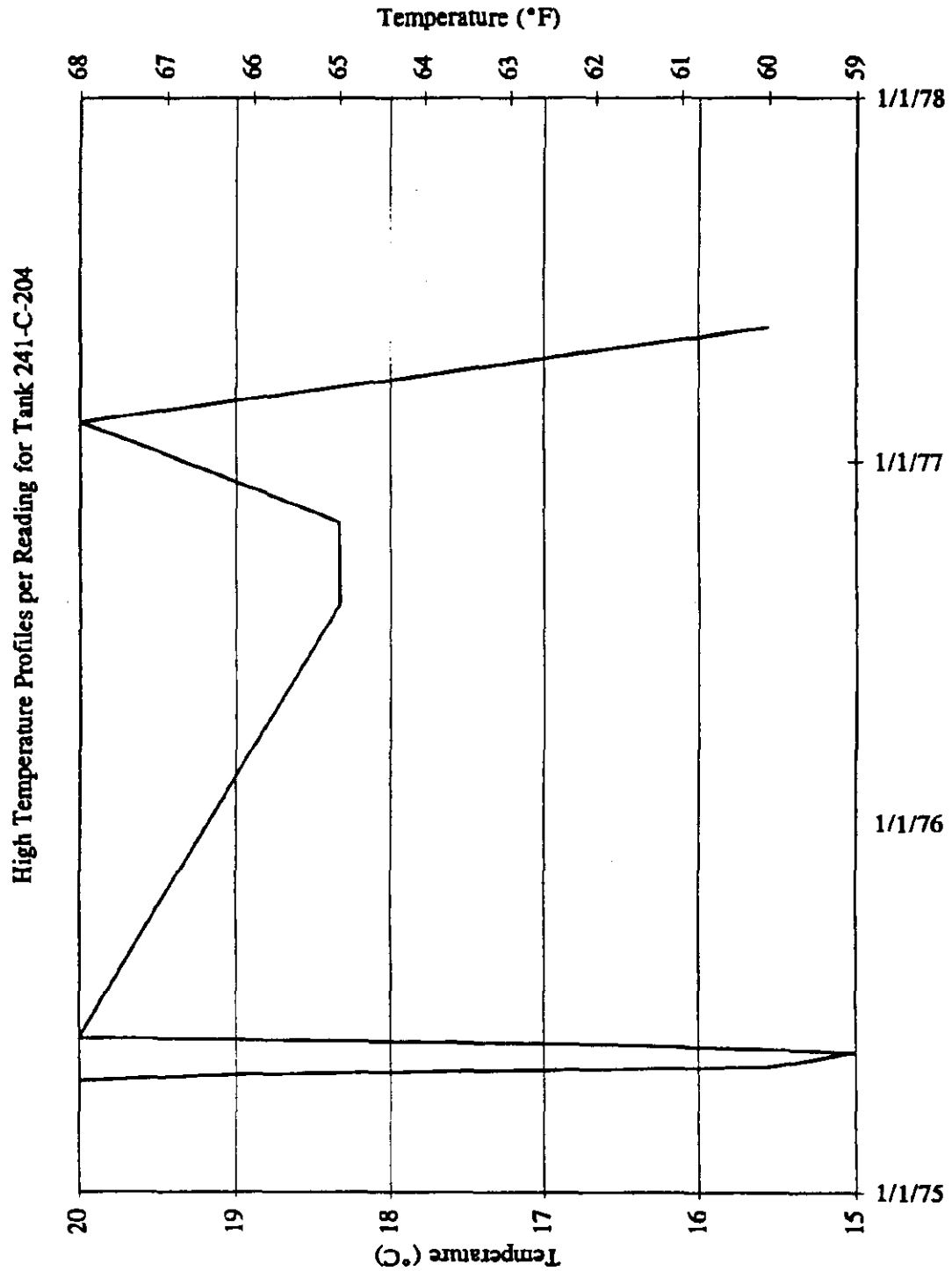
2.4.2 Internal Tank Temperatures

The thermocouple tree in tank 241-C-204 has produced little temperature data. Ten thermocouple tree readings from April 1975 through May 1977 were obtained from handwritten log sheets. After the last temperature reading on May 15, 1977, the log sheets indicated one of the following: the temperature information was not good, or the thermocouple tree was not there. The December 1986 photograph indicates that a thermocouple tree was not present at that time. Figure 2-6 shows the high temperature from each of 10 readings. For plots of the available thermocouple readings for tank 241-C-204, refer to Brevick et al. (1994b).

2.4.3 Tank 241-C-204 Photographs

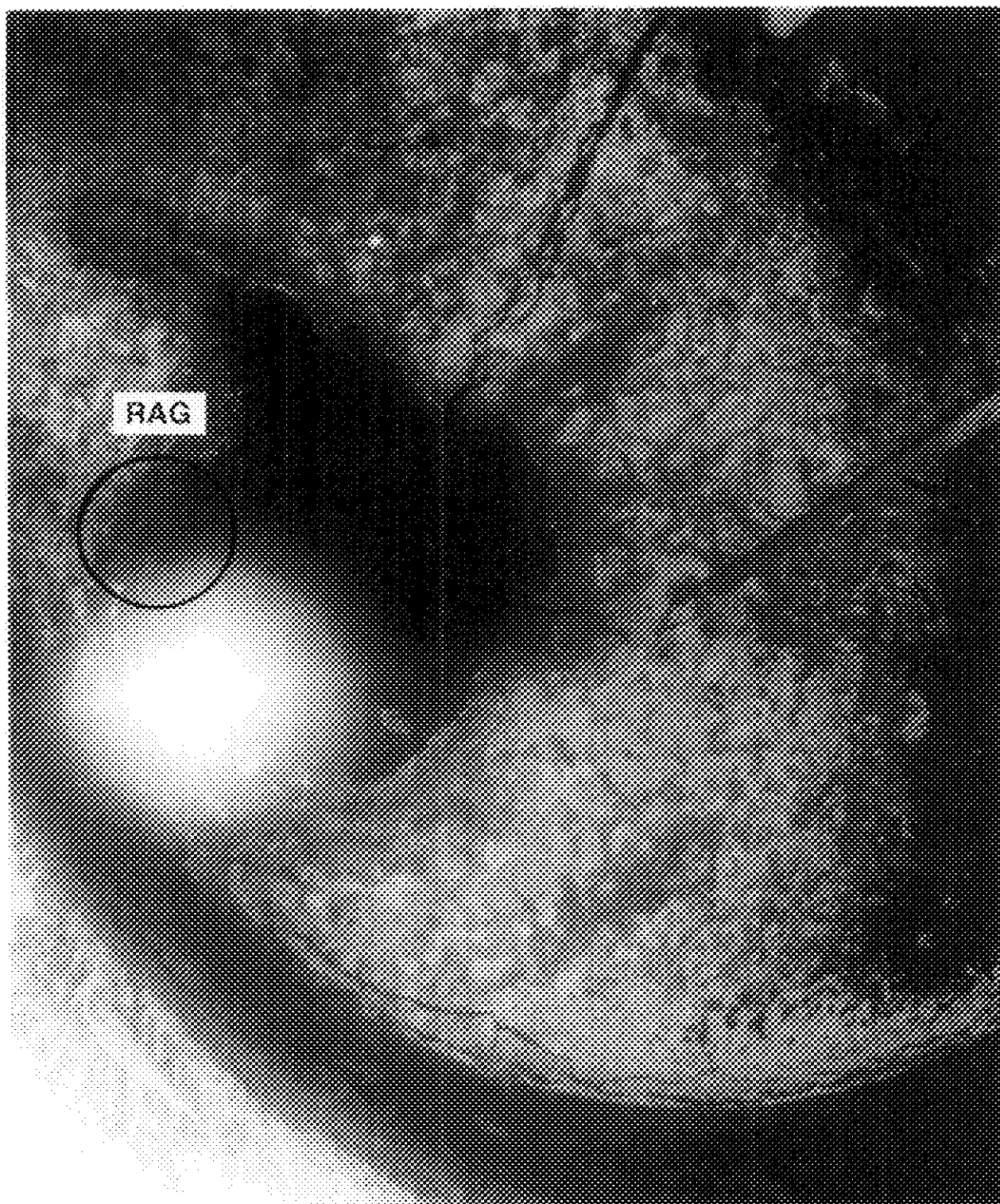
The 1986 photographs of the tank 241-C-204 interior shows a black and yellow mixed waste surface. The steel liner appears to be rusted or corroded. The equipment, which is clearly visible in the photographs, includes risers, a manual tape, a turbine pump, and a spare inlet nozzle. The missing thermocouple tree may also be present. Tank 241-C-204 has about 11 kL (3 kgal) of waste which equals approximately 46 cm (1.5 ft) of depth. Because no changes in the tank waste have occurred since the photographs were taken, they should represent the current tank contents. Figure 2-7 is an in-tank photograph of the surface of tank 241-C-204.

Figure 2-6. Tank 241-C-204 Weekly High Temperature Plot.



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Figure 2-7. In-Tank Photograph of the Surface of Tank 241-C-204.



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3.0 TANK SAMPLING OVERVIEW

Historically, single-shell tank waste samples have been analyzed to characterize the supernate, sludge, and/or saltcake in each tank. Data were compiled for samples obtained from the late 1950s to the present. The bulk of the data is from the auger samples taken in May 1995. Flammability screening of the tank headspace was conducted in 1996. Data also have been located for one sample from tank 241-C-204 which was received in the laboratory on June 26, 1975 (Wheeler 1975) and are shown in Appendix A. Information on a 1972 sample (Buckingham 1972) is also discussed. Table 3-1 summarizes the applicable DQOs and their respective analytical requirements. Results from tank headspace gas sampling will be provided in a revision to this report.

3.1 DESCRIPTION OF 1995 AUGER SAMPLING EVENT

This section describes the May 1995 auger sampling and analysis event for tank 241-C-204. Two auger samples were taken to satisfy the requirements of the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). The *Tank 241-C-204 Tank Characterization Plan* was written to direct and control the work (Schreiber 1995). This document was subsequently revised by Conner (1996a). For generic descriptions of the sampling and analytical procedures, refer to the *Tank Characterization Reference Guide* (DeLorenzo et al. 1994).

Two auger samples were taken from tank 241-C-204 in May 1995. Sample 95-AUG-022 was recovered from riser 7 (east coordinate) on May 2 with a 50 cm (20 in.) auger. This auger has 18 flutes: flute 1 begins at the auger shaft, and flute 18 ends at the tip. Sample 95-AUG-023 was recovered from riser 7 (west coordinate) on May 2 with a 50-cm (20-in.) auger (18 flutes). Riser 7 is 30 cm (12 in.) in diameter. Both samples were taken from this riser because no other risers were available for sampling. The flange used to bolt the auger sampler to the riser was rotated prior to taking sample 95-AUG-023. This led to the description of the samples as east or west coordinates of riser 7.

Both auger samples encountered a rag that was apparently on the waste surface directly under riser 7. Sampling information is shown in Table 3-2.

3.1.1 Sample Handling (1995 Auger Samples)

Auger samples 95-AUG-022 and 95-AUG-023 were received at the 222-S Laboratory on May 3, 1995 and were extruded on May 4 and 5, respectively. Both augers caught a rag which apparently had been dropped into the tank through riser 7. The extrusions were difficult because the rag became pinched between the auger and the auger sleeve. Photographs and a videotape of the extruded augers were taken. A photograph of an extruded auger (95-AUG-023) is shown in Figure 3-1.

Auger 95-AUG-022 was subsampled into half segments. The upper half consists of waste recovered on flute 8, and the lower half segment consists of waste material segregated from the rag (recovered from flutes 11 to 18). The rag material was placed in a separate jar. All other flutes were bare. The tank waste solids appeared dark brown. Auger recovery data are shown in Table 3-2.

Table 3-1. Data Quality Objective Requirements for Tank 241-C-204.

Sampling Event	Sampling Requirements	Applicable DQOs and Analytical Requirements
1996 Auger Sampling	Two full-depth profiles from widely spaced risers	<i>Safety Screening Data Quality Objective</i> (Babad and Redus 1994). Moisture content, energetics, total alpha activity. Total organic carbon and adiabatic calorimetry as secondary analyses.
1996 Tank headspace flammability screening	Flammability screen of tank vapor space	<i>Safety Screening Data Quality Objective</i> (Dukelow et al. 1995). Additional to requirements above for condensed phase samples (augers). Tank headspace flammability screening by combustible gas meter.
1996 Tank vapor space gas sampling ¹	Headspace gas sample and laboratory analysis	<i>Data Quality Objective for Tank Hazardous Vapor Safety Screening</i> (Osborne and Buckley 1995). Vapor sampling and analysis for positively and tentatively identified compounds (data not yet available).

Note:

¹Analysis is not complete.

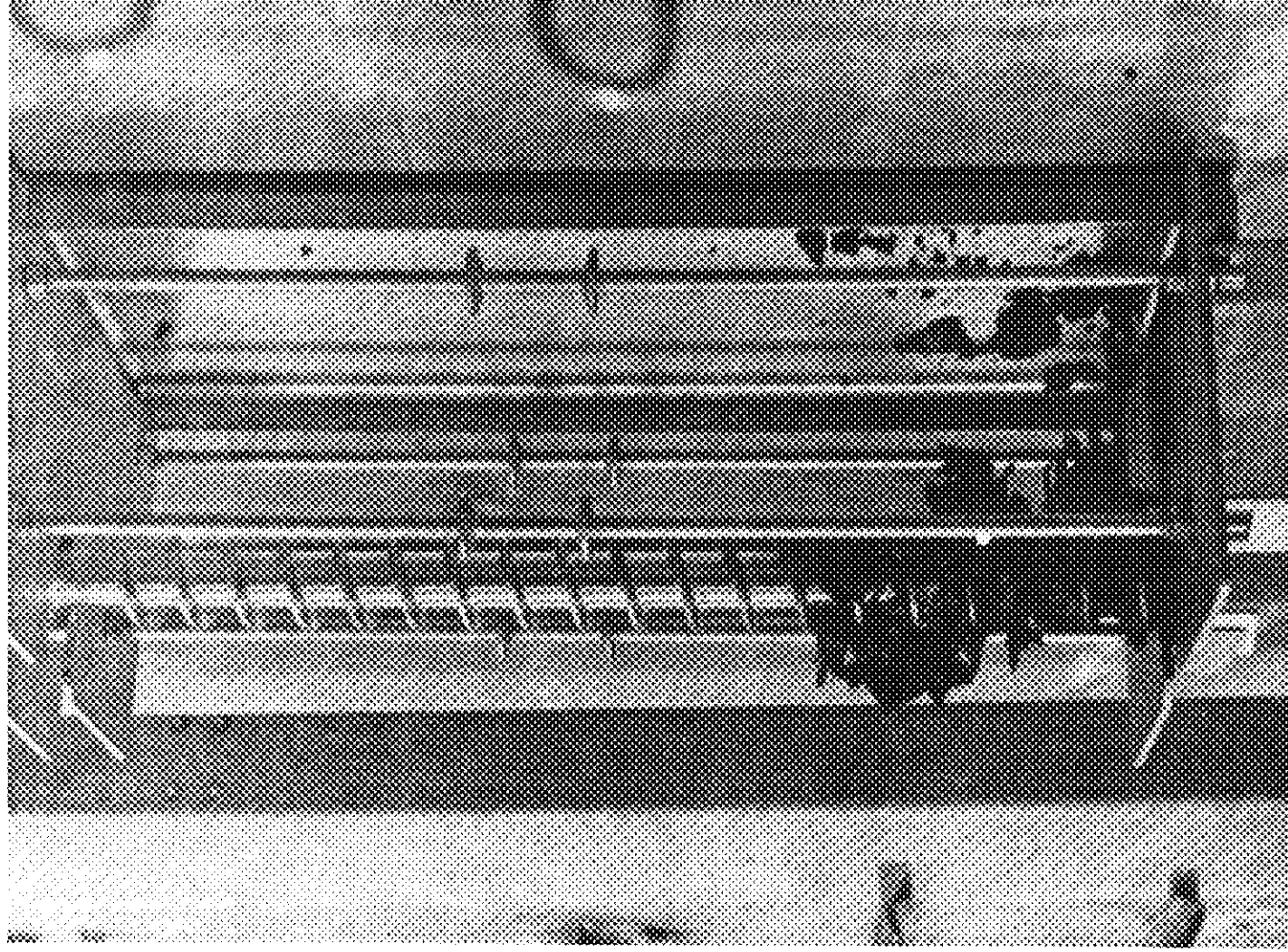
Table 3-2. 1995 Auger Sample Information.¹

Sample number	Expected sample length [cm (in.)]	Tank waste solids recovered (grams)	Rag material recovered (grams)	Drill string dose rate (mR/hr)
95-AUG-022	38 (15)	58.1	104.3	3.5
95-AUG-023	38 (15)	41.1	93.9	6

Note:

¹Couner (1993a)

Figure 3-1. Extrusion Photograph of Auger 95-AUG-023.



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For auger sample 95-AUG-023, tank waste solids were segregated from the rag material and subsampled as a whole segment (material was present only on flutes 13 to 18). The tank waste solids appeared to be a mixture of yellow and dark brown solids. The rag material was placed in a separate jar. After subsampling and incidental mixing, the material appeared brown.

3.1.2 Sample Analyses (1995)

After subsampling, safety screening analyses were performed. They were reported in *45-Day Safety Screen Results for Tank 241-C-204, Auger Samples 95-AUG-022 and 95-AUG-023* (Conner 1995a). Safety screening analyses for these tank waste solids consisted of DSC to evaluate fuel level and energetics potential, TGA to determine moisture content, and alpha proportional counting to determine total alpha activity. As the samples exceeded the screening limit for exothermic behavior by DSC, additional analyses were prescribed. The sample analysis plan (Schreiber 1995) was not strictly followed because the sample material remaining was limited. Deviations from Schreiber were documented and justified in Conner (1995a). At the request of the safety program, organic speciation was conducted at the Pacific Northwest National Laboratory (PNNL) 329 Laboratory.

The analytical procedures are listed in Table 3-3. The subsampling scheme is shown in Figure 3-2. Analytical results are described in Section 4.0.

3.2 TANK VAPOR FLAMMABILITY SCREENING

The flammability of the headspace in tank 241-C-204 was evaluated by combustible gas monitoring. The safety screening DQO notification limit for flammable gas concentration is 25 percent of the lower flammability limit (LFL) (Dukelow et al. 1995). The combustible gas meter used to sample the tank headspace reports results as a percent of the lower explosive limit (LEL). Because the National Fire Protection Association defines the terms LFL and LEL identically, the two terms are used interchangeably (NFPA 1995). The results indicate that there is no flammability concern for the tank headspace.

3.3 TANK VAPOR SAMPLING AND ANALYSIS

The tank headspace gases were sampled on July 3, 1996 to satisfy the *Data Quality Objective for Tank Hazardous Vapor Safety Screening* (Osborne and Buckley 1995). In situ vapor sampling was used to take samples for organic, inorganic, and radionuclide analyses. Results are not complete, and will be included in later revisions to this Tank Characterization Report.

Table 3-3. Analytical Procedures Used For Tank 241-C-204 Samples

Laboratory	Procedure Number	Title
WHC 222-S	LA-514-113	Differential Scanning Calorimetry (DSC)
WHC 222-S	LA-560-112	Determination of Percent Water as Weight Loss by Thermogravimetric Analysis (TGA) - Mettler ¹ TG 50
WHC 222-S	LA-508-101	Alpha and Beta in Liquid Samples
WHC 222-S	WHC-SD-WM-TP-104, Rev. 0 (Bechtold 1992)	Laboratory Test Plan for Adiabatic Calorimetry of Single-Shell and Double-Shell Tank Wastes
WHC 222-S	LA-342-100	Determination of Carbon by Hot Persulfate Oxidation and Coulometric Detection
PNNL 329	Not reported	Chromatography/mass spectrometry

Notes:

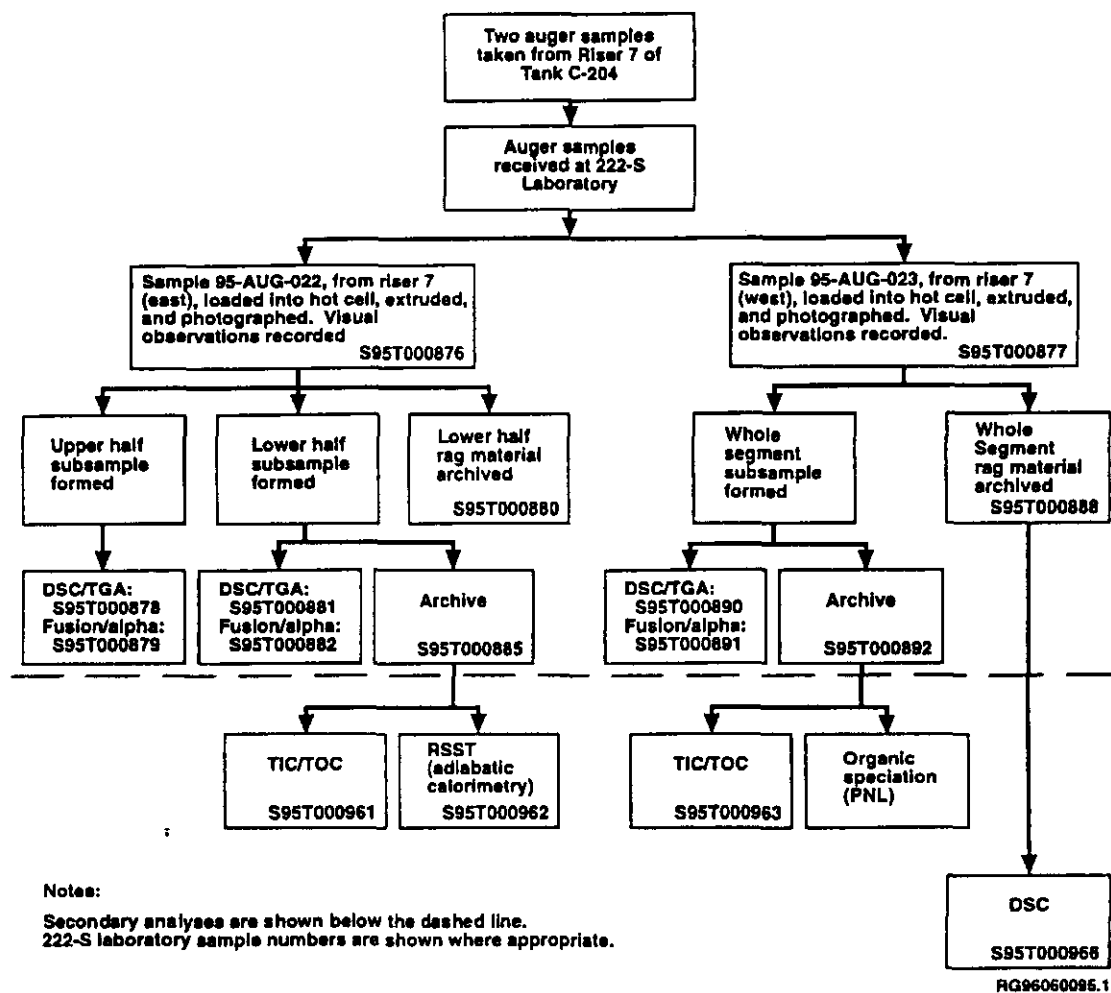
PNNL = Pacific Northwest National Laboratory
 WHC = Westinghouse Hanford Company

3.4 1972 HISTORICAL SAMPLING EVENT

A liquid sample was taken in 1972 and analyzed for exothermic characteristics by differential thermal analysis. The liquid waste in the tank was a candidate feed solution for in-tank solidification (a process used to concentrate certain aqueous wastes). A memo reporting this analysis (Buckingham 1972) noted "a strong exothermic reaction occurring around 150 °C...." When the sample was run on a different analyzer, a slight exothermic reaction was observed around 150 °C. Based on these qualitative results, it was recommended that the waste in tank 241-C-204 not be processed through in-tank solidification without further study. No information on sampling method, riser, nor sample depth was provided.

¹Mettler is a registered trademark of Mettler Electronics, Anaheim, California.

Figure 3-2. Laboratory Sample Handling Flowchart for Tank 241-C-204 Auger Samples.



3.5 1975 HISTORICAL SAMPLING EVENT

A liquid sample was received in the 222-S Laboratory in June, 1975, and analyzed (Wheeler 1975). Analysis by differential thermal analysis indicated no exotherm (temperature limit not given). The tank transfer history indicates that the vast majority of the tank liquids were pumped out in 1977 (Anderson 1990). The remaining wet solids probably bear little resemblance to the liquid analyzed in 1975. It is possible that the water-soluble constituents of the tank would be similar to this sample. No information on sampling method, riser, or sample depth was provided. The sample was a dilute solution (97 percent water). Pertinent data from this analysis is included in Appendix A. The data from this sample should be used with caution because the quality control criteria are not stated.

4.0 ANALYTICAL RESULTS AND WASTE INVENTORY ESTIMATES

This section describes the results associated with the May 1995 auger sampling of tank 241-C-204. The sampling and analysis were performed as directed in the sampling and analysis plan (SAP) (Schreiber 1995 and Conner 1996a). The SAP requirements were taken from the *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994). Analysis of the two augers was performed at the 222-S Laboratory. One subsample (Conner 1995b) was shipped to the Pacific Northwest National Laboratory 325 Laboratory for organic speciation at the request of the Organic Safety Program. (This particular sample was not covered under the quality assurance requirements of the SAP.) Table 4-1 shows the location of the analytical data tables.

Table 4-1. Data Tables for Tank 241-C-204.

Type of Data/Analysis	Table Location
Historical Tank Content Estimate	Table 2-5
Auger Sampling Information	Table 3-2
Chemical Summary	Table 4-2
Total Alpha (1995)	Table 4-3
Weight Percent Water (1995)	Table 4-4
Energetics (1995)	Table 4-5
DSC Exotherms (1995)	Table 4-6
TOC (1995)	Table 4-7
TOC, dry basis (1995)	Table 4-8
TIC (1995)	Table 4-9
Headspace Flammability	Table 4-10
Comparison of Analytical Data with Historical Estimates	Table 5-1
Comparison with Safety Screening Decision Criteria	Table 5-2

4.1 DATA PRESENTATION - AUGER SAMPLES

This section summarizes the analytical results from the 1995 auger sampling event for tank 241-C-204. The subsections below provide information derived from physical and chemical tests conducted on the samples. Data from the analyses of the auger samples were reported in the *Final Report for Tank 241-C-204, Auger Samples 95-AUG-022 and 95-AUG-023* (Conner 1996b).

4.1.1 Chemical Data Summary

Data from the two auger samples were combined to derive an overall concentration mean for each calculated analyte. In calculating the tank mean concentrations, each auger was weighted equally, that is, any subsamples from each auger were averaged together prior to calculating a simple average of each auger mean.

Table 4-2 shows the overall means. The first two columns of Table 4-2 contain the analyte and overall mean. The third column displays the relative standard deviation (RSD) of the mean. The RSD is defined as the standard deviation divided by the mean, multiplied by 100. The standard deviation of the mean was calculated using a nested random effects analysis of variance model using restricted maximum likelihood estimation techniques. The projected inventories listed in column 4 were calculated using the tank mass of 18,700 kg (see Table 2-4).

4.1.2 Radionuclide Data Summary: Total Alpha Activity

Analyses for total alpha activity were performed on three samples from tank 241-C-204 (upper and lower half segments from the riser 7, east auger sample and the whole segment from the riser 7, west auger sample). The samples were prepared by fusion using laboratory procedure LA-549-141, Rev. C-3 and were analyzed using laboratory procedure LA-508-101, Rev. D-2. The analyses were performed in duplicate.

Table 4-3 shows the total alpha activity taken from the 45-day report for tank 241-C-204 (Conner 1995a). The mean was calculated by weighting each auger equally. The total alpha tank inventory was calculated using the mean value for total alpha and a total solid waste weight of $1.92\text{E}+04$ kg (Brevick et al. 1994a).

4.1.3 Thermodynamic Analyses

Both TGA and differential scanning calorimetry DSC were performed on the auger samples from tank 241-C-204. These analyses were used to assess the moisture content, thermal stability, and reactivity of the samples. In addition, one sample was analyzed by adiabatic calorimetry to obtain a more accurate estimate of energetic potential.

Table 4-2. Chemical Summary for Tank 241-C-204.

Analyte	Overall Mean Concentration	Relative Standard Deviation of the Mean	Projected inventory
RADIONUCLIDES	$\mu\text{Ci/g}$	%	Cl
Total alpha	0.0322	60.5	0.602
CARBON	$\mu\text{g C/g}$		kg
TIC	10,500	11.9	196
TOC	126,000 (WHC) 60,000 ¹ (PNNL)	not reported	2,360 1,120
ORGANICS¹	$\mu\text{g/g}$		kg
Tributyl phosphate	330,000 ¹	not reported	1,190
Dibutyl phosphate	2,000 ¹	not reported	37
Monobutyl phosphate	none ¹	not reported	
Chelators, formate, acetate, oxalate, normal paraffin hydrocarbons, butyric acid, toluene, benzoic acid	trace ¹	not reported	trace
PHYSICAL PROPERTIES	%	%	kg
Percent Water	56.95	2.15	10,600
THERMODYNAMIC PROPERTIES	Joules/gram (dry basis)		
Energetics by DSC	Range: 813 to > 1234	Not applicable	Not applicable

Note:

¹These analyses were not conducted to the quality assurance requirements of the SAP and should only be used with caution.

Table 4-3. Tank 241-C-204 Analytical Results: Total Alpha Activity.

Sample Number ¹	Auger Portion	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)	Projected Inventory
		μCi/g	μCi/g	μCi/g	μCi/g	%	Ci
95-AUG-022					0.0322		0.602
0879	Upper half	0.00643	0.0145	0.0105 ^{2,3}			
0882	Lower half	0.0234	0.0121	0.0178 ³			
95-AUG-023							
0891	Whole	0.0511	0.0519	0.0515			

Notes:

¹Sample numbers begin with S95T00.²Spike recovery was below the 90 to 110 percent range specified in the SAP.³The RPD between the duplicate analyses was greater than the 10 percent limit specified in the SAP.

4.1.3.1 Thermogravimetric Analysis. Thermogravimetric analysis measures the mass of a sample while the temperature of the sample is increased at a constant rate. Nitrogen is passed over the sample during heating to provide an inert atmosphere. Any decrease in the weight of a sample during TGA analysis represents a loss of gaseous matter from the sample through evaporation or through a reaction that forms gas phase products.

The moisture content is estimated by assuming that all TGA sample weight loss up to a certain temperature, which can be as high as 200 to 250 °C, is caused by water evaporation. The temperature limit for moisture loss is chosen by the chemist or technician at an inflection point on the graph. Other volatile matter fractions can often be differentiated by inflection points as well. Figure B-1 shows a typical TGA scan for tank 241-C-204.

The tank 241-C-204 samples were analyzed by TGA using procedure LA-560-112, Rev. A-2. Analyses were performed in duplicate. A triplicate analysis was performed on sample S95T000878 because the relative percent difference (RPD) between the sample and duplicate results did not meet the criterion stated in the tank characterization plan. All results were well above the safety screening action limit of 17 percent water by weight. Table 4-4 summarizes the TGA results.

Table 4-4. Tank 241-C-204 Analytical Results: Weight Percent Water.

Sample Number	Auger Portion	Result		Duplicate		Sample Mean	Overall Mean	RSD (Mean)
		Weight % H ₂ O	Temperature Range (°C)	Weight % H ₂ O	Temperature Range (°C)	Weight % H ₂ O	Weight % H ₂ O	%
Auger 95-AUG-022								
S95T000878	Upper 1/2	58.32	30-200	50.44	30-220	56.08 ¹	56.95 ²	2.15
		TriPLICATE result		59.48	30-190			
S95T000881	Lower 1/2	55.02	30-220	56.39	30-210	55.70		
Auger 95-AUG-023								
S95T000890	Whole	59.92	30-220	56.08	30-220	58.00		

Notes:

¹The RPD between the original duplicate analyses was greater than the 10 percent limit specified in the SAP.

²The augers were equally weighted.

4.1.3.2 Differential Scanning Calorimetry. The DSC analysis is performed by heating the sample and increasing the temperature at a constant rate. Heat absorbed or emitted by the sample is measured as a function of time. Nitrogen is passed over the sample to remove any gases being released. The onset temperature for an endothermic or exothermic event is determined graphically. A typical DSC plot for the tank 241-C-204 samples is shown in Figure B-2.

The DSC analyses for tank 241-C-204 auger samples were performed using laboratory procedure LA-514-113, Rev. B-1. Table 4-5 summarizes the DSC results for the tank 241-C-204 auger samples. All samples exhibited large exotherms, and were run in triplicate because of poor reproducibility.

For samples S95T000878 and S95T000881, the DSC scans had not returned to baseline at the temperature limit of the test (that is, the exotherms were still progressing at 500 - 600 °C). The DSC analyzer can only integrate between fixed points on the graph; therefore, because the scans did not return to baseline, the data can only be reported as minimum values. The samples are stated as "greater than" values. No mean value was calculated for these two samples.

Table 4-5. Tank 241-C-204 Analytical Results: Energetics (wet weight basis).

Sample Number	Auger Portion	Run	Sample Weight	Transition 1		Transition 2		Transition 3	
			mg	Peak Temp. (°C)	ΔH (J/g)	Peak Temp. (°C)	ΔH (J/g)	Peak Temp. (°C)	ΔH (J/g)
Auger 95-AUG-022									
S95T000878	Upper half	1	22.05	106.8	551.4	289.4	1.4	448.6	> -195.7 ¹
		2	17.53	96.8	603.8	442.5	> -542.0 ¹	---	---
		3	35.63	162.5	451.1	282.7	> -7.0	505.9	> -305.9
S95T000881	Lower half	1	25.01	134.6	484.4	462.7	> -286.7 ¹	---	---
		2	25.53	102.8	583.8	341.6	> -33.4 ¹	---	---
		3	26.74	107.0	479.6	560.0	> -508.8	---	---
Auger 95-AUG-023									
S95T000890	Whole	1	25.21	132.6	422.2	450.7	-399.9 ²	---	---
		2	20.21	223.0	536.9	336.6	-279.6 ²	---	---
		3	25.72	136.5	460.2	452.6	-345.6	---	---
Pre-Dried Test Runs ³									
S95T000878 ³	Upper half	1	7.16	441.0	-1977	---	---	---	---
S95T000881 ³	Whole	1	12.91	547.8	-962.4	---	---	---	---

Notes:

¹Reproducible results for these samples were not obtained; therefore, RPDs were not calculated.

²The RPD between the original duplicate analyses was greater than the 10 percent limit specified in the sampling analysis plan.

³These samples were pre-dried, and the results are unofficial.

Table 4-6. DSC Exothermic Results and 95 Percent Confidence Interval Upper Limits.

Sample Number	Auger Portion	Run	Wet Wt. ΔH^1	Sample Wt. % Water	Dry Wt. ΔH	Mean	95% Confidence Interval of the Mean Upper Limits
			J/g	%	J/g	J/g	J/g
Auger 95-AUG-022							
S95T000878	Upper half	1	> -195.7 ²	56.08	> -445.6	n/a	n/a
		2	> -542.0		> -1,234		
		3	> -7.0		> -16		
			> -305.9 ²		> -696.5		
S95T000881	Lower half	1	> -286.7	55.70	> -647.3	n/a	n/a
		2	> -33.4		> -76.1		
		3	> -508.8		> -1,149		
Auger 95-AUG-023							
S95T000890	Whole	1	-399.9	58.00	-952.1	-813.6	-1055
		2	-279.6		-665.7		
		3	-345.6		-822.9		
Pre-Dried Test Runs							
S95T000878 ³	Upper half	1	n/a	56.08	> -1,977	n/a	n/a
S95T000881 ³	Whole	1	n/a	55.70	> -962.4	n/a	n/a

Notes:

n/a = not applicable

 ΔH = change in enthalpy¹All exothermic reactions occurred in the second transition unless otherwise noted.²The exothermic reaction for this sample came from the third transition.³These samples were pre-dried, and the results are unofficial.

The endotherms for the samples were quite large and dominated the scans as far as 300 °C. In an attempt to isolate the exotherms, subsamples from S95T000878 and S95T000881 were preheated to approximately 240 °C by TGA to remove water from the samples. The temperature was raised at a rate of 10 °C per minute. The subsamples then were analyzed by DSC. These runs, which are marked as "test," are unofficial and can only be considered approximate results.

In addition to the DSC analyses conducted on the tank waste material, a subsample of the rag captured with auger sample 95-AUG-023 was analyzed by DSC. The results were inconclusive. The thermogram (see Figure B-3) did exhibit an upward (exothermic) trend, but no distinct exotherms could be calculated.

4.1.3.3 Adiabatic Calorimetry (Reactive System Screening Tool). If a sample exceeds the screening limit for energetics by DSC, the safety screening DQO (Babad and Redus 1994) calls for secondary analyses. One of these analyses is the Reactive System Screening Tool. This analysis attempts to maintain adiabatic conditions by adding heat to a bomb calorimeter to compensate for sample heat losses. Approximately 10 cm³ of dried, pulverized sample is placed in the calorimeter. A heater heats the sample at a slow, constant rate until self heating from the exothermic reactions dominates the temperature history, and the reaction goes to completion. By measuring the rate of temperature change, the total temperature change, and the heat capacity of the sample, the chemical rate kinetics and total exothermic energy can be calculated.

The archive sample from auger 95-AUG-022 was prepared for adiabatic calorimetry by removing the jar lid and allowing the sample to dry in the hot cell for several weeks (from 57 percent to 26 percent moisture). Some additional (partially decomposed) rag material was discarded, leaving brown granular solids. Of this material, 8.84 g were loaded into the calorimeter and subjected to the Reactive System Screening Tool analysis under 7 bar-gauge nitrogen. Results are reported in Conner and Bechtold (1995) and summarized in Appendix B. The results indicate that the waste cannot support a propagating combustion. The self-heating response was complex and sluggish and never exceeded 6 °C per minute (uncorrected) with a total of 0.0018 moles/gram of noncondensable gas evolved. Two exothermic events were detected: from 160 to 260 °C and from 280 to above 400 °C. The second exotherm was interrupted by a sharp endotherm at 300 °C. This is consistent with the decomposition of tributyl phosphate in simulant experiments (Cowley and Postma 1996). At 435 °C, the self-heating rate plummeted because of complete reactant exhaustion before the heater was turned off at 449 °C. The sample material had been reduced to a black, unfused, granular material similar to charcoal. This indicates that the sample was fuel-rich, which is consistent with the finding of tributyl phosphate (see Section 4.1.5). The final weight of the sample was 7.44 g indicating a 15.8 percent reduction in mass. A moderate amount of condensate was generated which felt lubricative to the touch and perhaps indicated an organic component. Graphs of temperature versus time and change-in-temperature with time for the test are provided in Appendix B. The second graph provides a good illustration of the two exotherms. Values for the heating rate (J/g/°C) for each exotherm and total energy released (J/g) are also presented but as a function of the heat capacity of the sample

(which was not determined). The maximum heating rate and total energy released may be calculated by assuming a heat capacity value for the sample.

4.1.4 Carbon Analyses

As directed in Schreiber (1995), TOC analyses were requested once the exothermic nature of the samples was identified by DSC. The TIC may be determined incidentally with the TOC analyses; therefore, TIC analyses were performed and reported along with TOC.

4.1.4.1 Total Organic Carbon. Two samples were submitted for TOC analysis: the lower half subsample from 95-AUG-022 (riser 7, east) and the whole segment sample from 95-AUG-023 (riser 7, west). Normally, each sample that exceeded the action limit for energetics would be submitted for TOC analysis, however, the upper half subsample from 95-AUG-022 (riser 7, east) was completely used up during the primary safety screening analyses. The TOC results are shown in Tables 4-7 and 4-8.

Table 4-7. Tank 241-C-204 Analytical Results: Total Organic Carbon.

Sample Number ¹	Auger Portion	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)	Projected Inventory
		µg C/g	µg C/g	µg C/g	µg C/g	%	kg C
95-AUG-022					1.26E+05	10.3	2,360
Not applicable	Upper half	Not requested	Not requested	Not requested			
0961	Lower half	1.48E+05	1.30E+05	1.39E+05 ^{2,3}			
95-AUG-023					1.26E+05	10.3	2,360
0963	Whole	91,800	1.44E+05	1.13E+05 ³			
		Triplicate result	1.03E+05				

Notes:

¹Sample numbers begin with S95T00.

²The spike recovery was greater than the 90 to 110 percent range specified in the SAP.

³The RPD between the duplicate analyses was greater than the 10 percent limit specified the SAP.

Table 4-8. Adjusted Dry Total Organic Carbon Results.

Sample Number ¹	Auger	Auger Portion	Sample Mean	Sample Weight % Water	Adjusted Dry Mean Result
			g C/g	%	µg C/g
0961	95-AUG-022	Lower half	1.39E+05	55.70	3.14E+05
0963	95-AUG-023	Whole	1.13E+05	58.00	2.69E+05

Note:

¹Sample numbers begin with S95T00.

4.1.4.2 Total Inorganic Carbon. The only inorganic analysis performed on tank 241-C-204 auger samples was TIC. This analysis was not required, but it was performed incidentally with the TOC analysis. The carbonate (inorganic carbon) component of the sample must be removed before the organic carbon content can be determined. The TIC results are shown in Table 4-9. The spike recovery for TIC was low (see Section 5.1.2). Because TIC results were not called out in the tank characterization plan, no attempt was made to improve the spike recovery. These results should be used with caution.

Table 4-9. Tank 241-C-204 Analytical Results: Total Inorganic Carbon.

Sample Number ¹	Auger Portion	Result	Duplicate	Sample Mean	Overall Mean	RSD (Mean)	Projected Inventory
		µg C/g	µg C/g	µg C/g	µg C/g	%	kg C
95-AUG-022					10,500	16.9	196
Not applicable	Upper half	Not requested	Not requested	Not requested			
0961	Lower half	9,360	8,130	8,740 ^{2,3}			
95-AUG-023					10,500	16.9	196
0963	Whole	13,800	10,700	12,300 ³			
		Triplicate result	12,400				

Notes:

¹Sample numbers begin with S95T00.²Spike recovery was below the 90 to 110 percent range specified in the SAP.³The RPD between the duplicate analyses was greater than the 10 percent limit specified in the SAP.

4.1.5 Organic Analyses

Once the high TOC numbers were returned, safety program personnel were consulted. At the direction of safety personnel, the remaining sample from 95-AUG-023 (riser 7, west whole segment subsample) was submitted to the Pacific Northwest National Laboratory (329) for organic speciation by gas chromatography/mass spectrometry. This analysis was focused on organic species commonly found in Hanford waste streams (normal paraffin hydrocarbons, low molecular weight acids, chelators, and tributyl phosphate). The sample was submitted to the Pacific Northwest National Laboratory with a letter of instruction (Conner 1995b) at the direction of the Organic Safety Program. Results indicated the sample contained fully 33 percent tributyl phosphate by weight with a minor amount of dibutyl phosphate. Other organic compounds were found in only trace quantities. As the quality assurance protocol for this effort was not the same as specified in the sampling and analysis plan, the data should be used with caution. For summary data, see Table 4-2.

4.1.6 Flammability Screening

The tank headspace was screened for flammability concerns (Dukelow et al. 1995). The results indicate the tank headspace is below the level of concern (0 percent of the LFL) (see Table 4-10).

Table 4-10. Results of Combustible Gas Meter Monitoring of the Headspace of Tank 241-C-204 on June 3, 1996.

Percent of lower flammability limit	0
Oxygen (%)	20.7
TOC (ppm)	13.7
Ammonia (ppm)	0

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5.0 INTERPRETATION OF CHARACTERIZATION RESULTS

This section evaluates the overall quality and consistency of the available results for tank 241-C-204, and it assesses and compares the results against historical information and program requirements. The assessment of the tank profile is limited because of the small data set required for safety screening and problems with sample recovery.

5.1 ASSESSMENT OF SAMPLING AND ANALYTICAL RESULTS

This section evaluates sampling and analysis factors that may impact data interpretation. These factors are used to assess the quality and consistency of data and to identify any limitations in the data use.

Because of the small number of analyses conducted on tank 241-C-204 auger samples and the problems encountered in sampling, information is limited. The intent of the sampling event was to screen the tank for safety issues. A safety issue was identified (exothermic potential), and secondary analyses were conducted. No statements regarding the homogeneity of tank contents can be inferred as both samples were recovered from the same tank access point (riser 7) and were contaminated with rag material. Nevertheless, the information was useful, and some qualified statements regarding tank contents can be made. Results indicate that an exothermic event is not possible, because of the sluggish reaction of the waste and high moisture content.

5.1.1 Field/Laboratory Observations

Both augers encountered a rag in the tank. For auger sample 95-AUG-022, field personnel reported hitting a very hard layer near the projected tank bottom. Because depth measurements are not always exact, the sampling crew concluded that the tank bottom may have been reached. Augering was stopped, and the sample was extracted. For auger sample 95-AUG-023, a similar hard layer was encountered four inches higher than the previous sample. In retrospect, the tank bottom may not have been reached on either sample, and augering may have been hindered because the rag was bound up in the auger. Nevertheless, sufficient tank waste material was recovered to perform safety screening analyses according to the SAP (Schreiber 1995).

Extrusion of both augers was difficult because of the rag which was jammed between the auger and the auger sleeve. The rag caught by both augers is visible in in-tank photographs (see Figure 5-1). Upon extrusion, hot cell technicians were directed to segregate the rag material from the tank waste material. No rag fibers were visible in the segregated tank waste. Subsequently, hot cell chemists reported seeing rag fibers in the sample air-dried in preparation for adiabatic calorimetry. Even after several weeks of drying, the sample

appeared wet and had a gummy consistency, perhaps indicating a high organic content. Visible fibers were removed before the analysis.

No other chemist reported seeing rag fibers. This is significant because in the other analyses, the subsamples are quite small (approximately 25 mg for DSC and TGA runs and 0.5 g to 1.0 g for a TOC analysis or fusion). For these small sample sizes, the absence of visible fibers does not rule out the possibility of contamination (on the rag material or the material itself), but it does indicate that large exotherms and high TOC results are largely attributable to organics in the tank waste. However, contamination from the rag material cannot be ruled out.

The conclusion from adiabatic calorimetry is that the sample would not support a propagating combustion reaction. This result, coupled with the high moisture content of the samples (55 percent), rules out any imminent concern. Nevertheless, the difficulties in sampling (that is, both samples were from the same riser, encountered the rag, and probably didn't reach the tank bottom) and unexpected and unusual analytical results (that is, high DSC, TOC, and tributyl phosphate results) lead to the conclusion that further sampling and analysis is warranted. The original SAP (Schreiber 1995) indicated that another riser could be made available for sampling--with some effort.

5.1.2 Quality Control Assessment

The usual quality control assessment includes an evaluation of the appropriate standard recoveries, matrix spike recoveries, duplicate analyses, and blanks that are performed in conjunction with the chemical analyses. All the pertinent quality control tests were conducted on the 1995 auger samples, allowing a full assessment regarding the accuracy and precision of the data. The specific criteria for all quality control checks were given in the SAP (Schreiber 1995, Conner 1996a). Quality control results outside these criteria were identified in the data summary tables (see Section 4.0).

The standard and matrix spike recovery results provide an estimate of the accuracy of the analysis. If a standard or spike recovery is above or below the given criterion, the analytical results may be biased high or low, respectively. All standard recoveries were within the defined criterion. The single matrix spike recovery for total alpha activity was below the 90 to 110 percent criterion (61.9 percent recovery). This may have been caused by low sample activities and self-shielding. The single matrix spike recoveries for TIC and TOC were also outside the criterion.

Analytical precision is estimated by the relative percent difference (RPD), which is defined as the absolute value of the difference between the primary and duplicate samples, divided by their mean, times one hundred. The SAP criterion for analytical precision is ≤ 10 percent for all analytes. Total alpha activity had two of three RPDs outside this limit. Considering that no result was more than 10 times the detection limit, some variability is expected. Because all results were below the decision threshold of 41 $\mu\text{Ci/g}$ by a factor of 800 or

more, reruns were not considered necessary (Conner 1995a). One of three TGA samples had an RPD above the ≤ 10 percent SAP limit. Both the TIC and TOC RPDs were above the criterion as well as the single DSC sample for which an RPD was calculable. The high RPD result for the DSC analyses was probably caused by the unusual exothermic behavior and the small sample size. The poor reproducibility of DSC and TOC results may have been caused by the extraordinarily high organic content or contamination by a rag that was extruded with the samples (leading to poor homogenization).

5.1.3 Data Consistency Checks

Comparing different analytical methods is helpful when assessing data consistency and quality. Because of the limited data, few such checks can be made. The only comparison provided here is to compare the organic carbon results from the Westinghouse Hanford Company and the Pacific Northwest National Laboratory with the organic speciation results from the Pacific Northwest National Laboratory.

The TOC results of approximately 13 percent, which were generated at the 222-S laboratory, are over twice the TOC value of six percent which was generated by the PNNL. This discrepancy may be due to sample inhomogeneity or differences in the analytical procedures. The tributyl phosphate results of 33 percent (equivalent to 18 percent TOC) determined by gas chromatography/mass spectrometry are well above either reported TOC value. However, Mong and Campbell (Conner 1996b) note that the persulfate oxidation method used to generate the TOC data does not give complete nor quantitative results for tributyl phosphate. Therefore, the TBP results are not inconsistent with the lower TOC results.

5.2 COMPARISON OF HISTORICAL AND RECENT ANALYTICAL RESULTS

Because the 1972 and 1975 samples were liquids and the tank now contains only solids, no extensive comparison of results is attempted. It is interesting to note that exotherms were detected in the 1972 sample and in the 1995 augers, but they were not detected in the dilute 1975 sample.

5.3 COMPARISON OF ANALYTICAL AND TRANSFER DATA

The HTCE predictions for the contents of tank 241-C-204, taken from *Hanford Tank Chemical and Radionuclide Inventories: HDW Model Rev. 3* (Agnew et al. 1996), are shown in Table 5-1 with the analytical results from the 1995 auger sampling event. Because the HTCE values have not been validated, the comparison is for information only.

Comparisons were possible for only four analytes. The total inorganic carbon comparison demonstrated the best agreement. The water content predictions agreed moderately, but there was poor agreement between the two data sets for total alpha activity and extremely

poor agreement for total organic carbon. Reasons for the discrepancies could include the following: high variability in the waste stream modeled, incorrect modeling assumptions, or sampling complications such as the rag.

Table 5-1. Comparison of HTCE Predictions with the 1995 Analytical Results.

Analyte	HTCE Estimate ¹	1995 Analytical Result ²	Relative Percent Difference
Total alpha activity	0.00263 $\mu\text{Ci/g}^3$	0.0322 $\mu\text{Ci/g}$	170%
TOC	4,120 $\mu\text{g C/g}$	1.26E+05 $\mu\text{g C/g}$	Not applicable
TIC	9,180 $\mu\text{g C/g}$	10,500 $\mu\text{g C/g}$	13.4%
Water	44.1 wt. %	56.94 wt. %	25.4%

Notes:

¹Agnew et al. (1996)

²Conner (1996b)

³The result is based only on a plutonium estimate.

5.4 EVALUATION OF PROGRAM REQUIREMENTS

The two auger samples taken from tank 241-C-204 in 1995 were acquired to meet the requirements of the original release of the safety screening DQO (Babad and Redus 1994). The headspace flammability screening conducted in 1996 was performed to satisfy a later version of the DQO (Dukelow et al. 1995). A vapor sample was taken in June 1996 to comply with the requirements of (Osborne et al. 1995). As analyses are not yet completed, these results will be addressed in a revision to this tank characterization report. Only safety screening issues are addressed at this time.

Data criteria in the original safety screening DQO (Babad and Redus 1994) are used to assess waste safety and to check for unidentified safety issues. The sampling requirement of the DQO was to recover vertical profiles of waste from two widely spaced risers. The SAP (Schreiber 1995 and Conner 1996a) stated that although a second riser could be used with considerable effort, only one 12-in. riser was readily available for sampling. The SAP says "Discussions with personnel in the tank waste remediation system indicated that since samples out of both risers offered a separation of only two feet, in this case it was acceptable to take two samples out of the same riser, offering a separation of approximately 10 inches." It is worth noting that tank 241-C-204 is only 6.1 m (20 ft) in diameter, not 22.9 m (75 ft) in diameter as are most other waste tanks. Because both auger samples were from the same riser and both hit a rag on the surface of the waste and may not have retrieved a full length sample, it is judged that the sampling requirements of the safety screening DQO were not met. A sample from a second riser is recommended.

The analytical requirements of the DQO were to evaluate energetic potential by DSC and TGA and to evaluate the criticality potential by total alpha counting (see Table 5-2). All samples submitted for DSC exceeded the -481 J/g action limit. This triggered secondary analyses consisting of TOC on two of three subsamples (one sample was completely used up during primary analyses) and adiabatic calorimetry on one of two archive samples. The TOC results were far over the action limit of 30,000 $\mu\text{g/g}$.

The SAP called for cyanide analyses once a sample exceeded the safety screening limit for energetics by DSC. However, cyanide analyses were not run for samples from tank 241-C-204 even though the safety screening limits for energetics were exceeded. Safety program personnel directed that the limited archive material be saved for more appropriate secondary analyses. Once the exothermic nature of the tank 241-C-204 auger samples was detected by DSC analyses, the transfer history of the tank was reviewed to identify the responsible waste stream. Transfers from the hot semiworks process (DeLorenzo et al. 1994) are suspected to be the source of the organics-rich waste found in tank 241-C-204. These waste streams and other streams directed to the tank do not contain ferrocyanide, the cyanide parent chemical of concern to the safety program. Therefore, analyzing tank 241-C-204 samples for cyanide was determined to be of little value.

The flammability issue of the DQO was met by combustible gas monitoring. Results indicate no flammability concern exists (0 percent of LFL).

The analytical requirements of the DQO were met. Although the DSC and TOC action limits were exceeded, the moisture content of 55 percent is well above the 20 percent level necessary for mitigation of an exothermic event (Dukelow et al. 1995). In addition, the results of adiabatic calorimetry indicate that the sample will not propagate a reaction. None of the analytical data indicate that the tank is unsafe according to the criteria in the DQO (Babad and Redus 1994 and Dukelow et al. 1995). However, the difficulties in sampling (that is, both samples were from the same riser, encountered the rag, and probably didn't reach the tank bottom) and unexpected and unusual analytical results (that is, high DSC, TOC, and tributyl phosphate) lead to the conclusion that further sampling and analysis is warranted.

Table 5-2. Comparison of 1995 Auger Sample Data to Safety Screening DQO Decision Criteria.

Decision Variable	Decision Criteria Threshold (Action Limit)	Number of Subsamples Outside Threshold/Total Subsamples
Total fuel content	> 481 J/g	3/3
Percent water	< 17 weight percent	0/3
Total alpha	> 41 $\mu\text{Ci/g}^1$	0/3
TOC	> 30,000 $\mu\text{g/g}$	2/2
Combustible gas meter	< 10 percent of LFL	0/1

Note:

¹Derived from a specification of 1 g/L, assuming a waste density of 1.5 g/cm³.

6.0 CONCLUSIONS AND RECOMMENDATIONS

The waste in tank 241-C-204 was auger sampled in May, 1995, and the tank headspace was screened for flammable gases in June, 1996. The *Tank Safety Screening Data Quality Objective* (Babad and Redus 1994 and Dukelow et al, 1995) governed the sampling and analysis of samples 95-AUG-022 and 95-AUG-023 and the flammability screening event.

The auger sampling event was hindered because both augers encountered a rag in the tank. This hindered sample recovery and probably meant that the tank bottom was not reached. The analytical results on the retrieved waste indicate unexpectedly high energetics and TOC. Exotherms in excess of -1,234 J/g (dry basis) were detected (final values could not be obtained because the exotherms were still progressing at the temperature limit of the test, that is, 500 or 600 °C). The action limit was -481 J/g. The TOC results were approximately 13 percent (26 percent dry basis). Results of organic speciation suggest that the organic component is almost exclusively tributyl phosphate. The moisture content of the samples was 57 percent by TGA, and the total alpha concentration averaged 0.0322 $\mu\text{Ci/g}$, well below the action limit of 41 $\mu\text{Ci/g}$.

Although the DSC results exceeded the safety action limit, TGA results indicate the tank has sufficient moisture to mitigate any exothermic event. Further, the adiabatic calorimetry results indicate the sample will not propagate an exothermic reaction.

Headspace flammability is not a concern because the results of combustible gas monitoring indicate headspace gases are at 0 percent of the LFL.

Because the sampling event was hindered by the presence of the rag, and the tank bottom of the tank was probably not reached, resampling from another riser is recommended.

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APPENDIX A

1975 GRAB SAMPLE RESULTS

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APPENDIX A

1975 GRAB SAMPLE RESULTS

The data from this sample is provided for historical information and should be used with caution. The tank transfer history indicates that the majority of tank 241-C-204 liquids were pumped out in 1977 (Anderson 1990). The remaining wet solids may bear little resemblance to the liquid analyzed in 1975.

Table A-1. Supernatant Sample (#T-5625) from Tank 241-C-204.¹

Waste Tank 241-C-204 October 23, 1975 Received June 26, 1975		
Component	Lab Value	Lab Units
Physical Data		
Visual observations and over-the-top reading	Pale Yellow, <1% solids, 3 rad/hr	
pH	9.1	
Specific gravity	0.9974	
Differential thermal analysis	No exotherm	
Percent H ₂ O	97.33%	
Cooling Curve		
Temperature	Time	Solids
35 °C	45 minutes	No solids
30 °C	45 minutes	No solids
25 °C	45 minutes	No solids
20 °C	45 minutes	No solids
15 °C	45 minutes	No solids
10 °C	45 minutes	No solids
5 °C	30 minutes	No solids
Chemical Analysis		
OH	< 1.89E-02	M
Al	1.92E-03	M
Na	0.446	M
NO ₂	8.55E-02	M
NO ₃	0.171	M
SO ₄	Canceled	M
PO ₄	4.28E-03	M
F	5.98E-05	M
Cl	< 7.52E-03	M
CO ₃	0.115	M
Radiological Analysis		
Pu	< 4.44E-06	g/gal
GEA: ¹³⁷ Cs	1.79E+03	μCi/gal
^{89/90} Sr	14.86	μCi/L

Notes:

¹Wheeler (1975)

APPENDIX B

SELECTED THERMODYNAMIC DATA

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Figure B-1. Typical Thermogravimetric Analysis Scan.

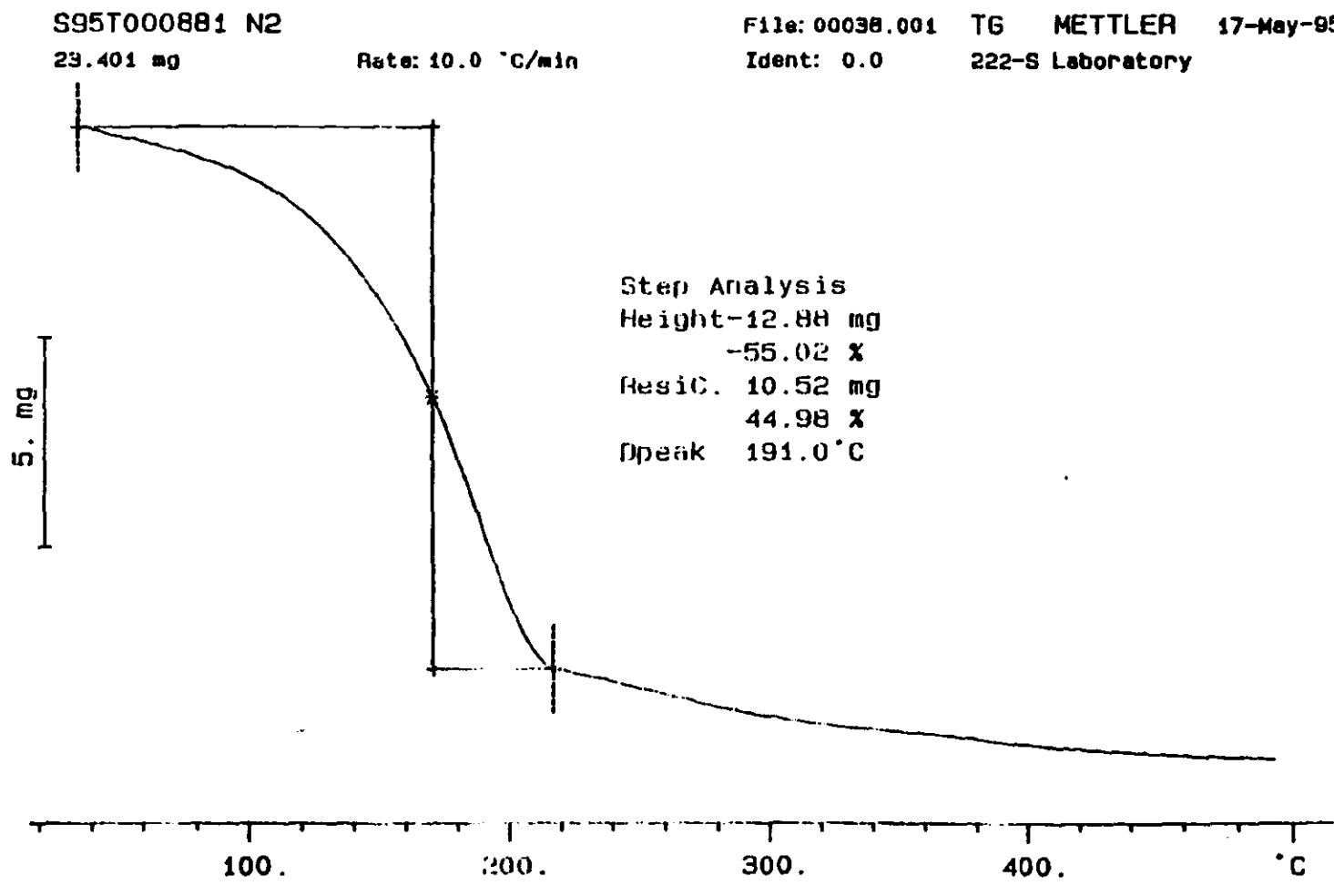
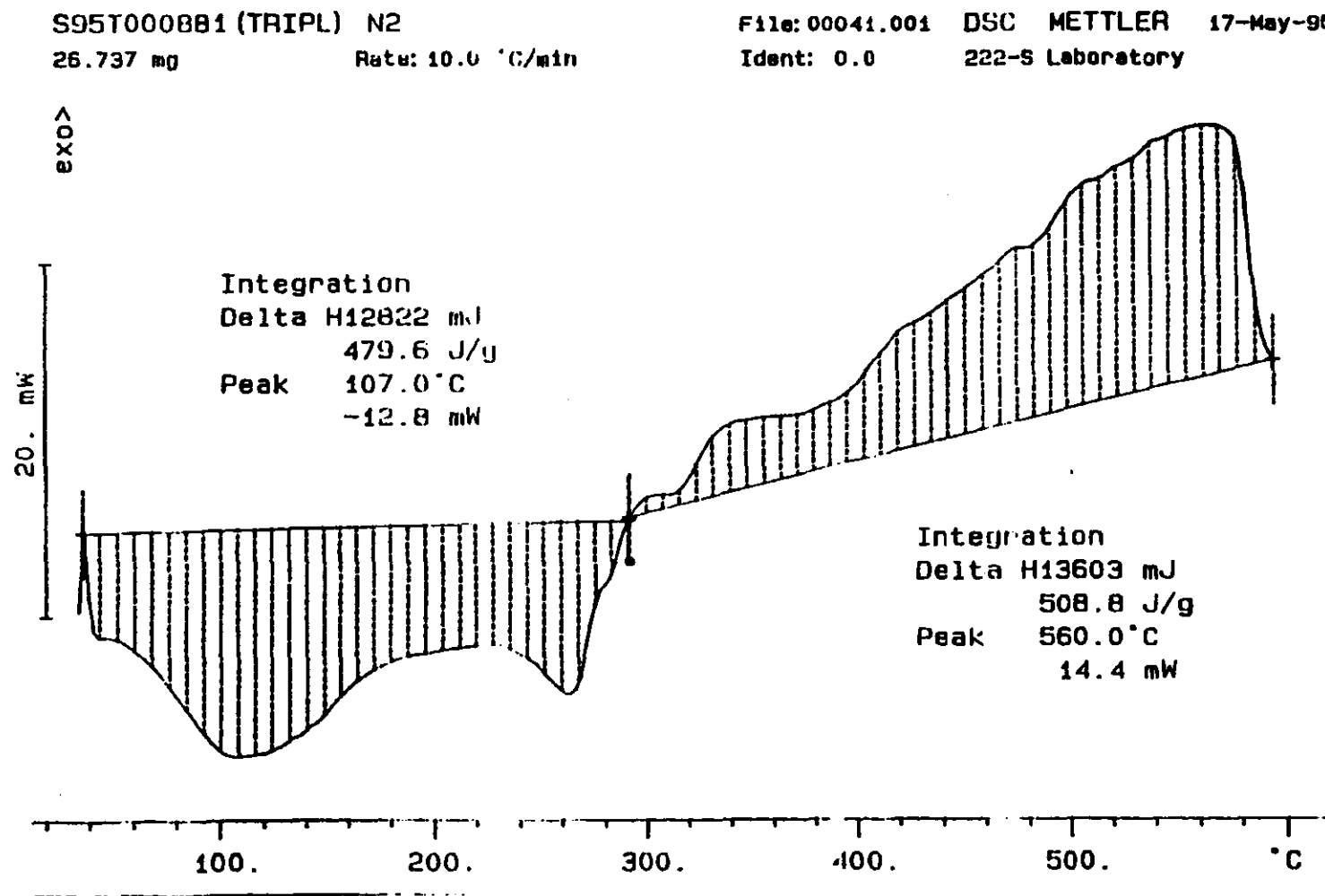


Figure B-2. Typical Differential Scanning Calorimetry Scan.



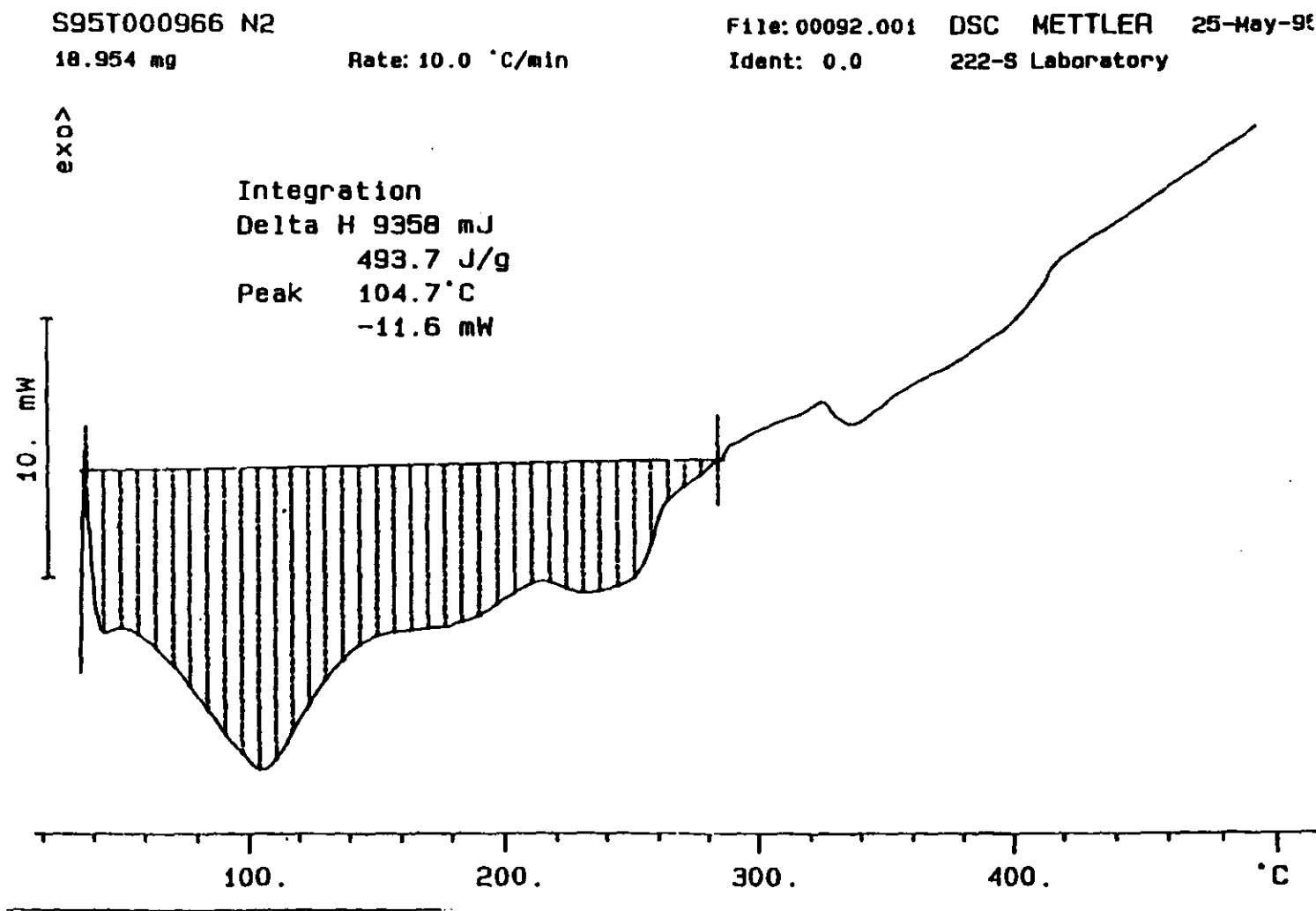


Figure B-3. Differential Scanning Calorimetry Rag Scan.

Figure B-4. Adiabatic Calorimetry: Temperature vs Time.

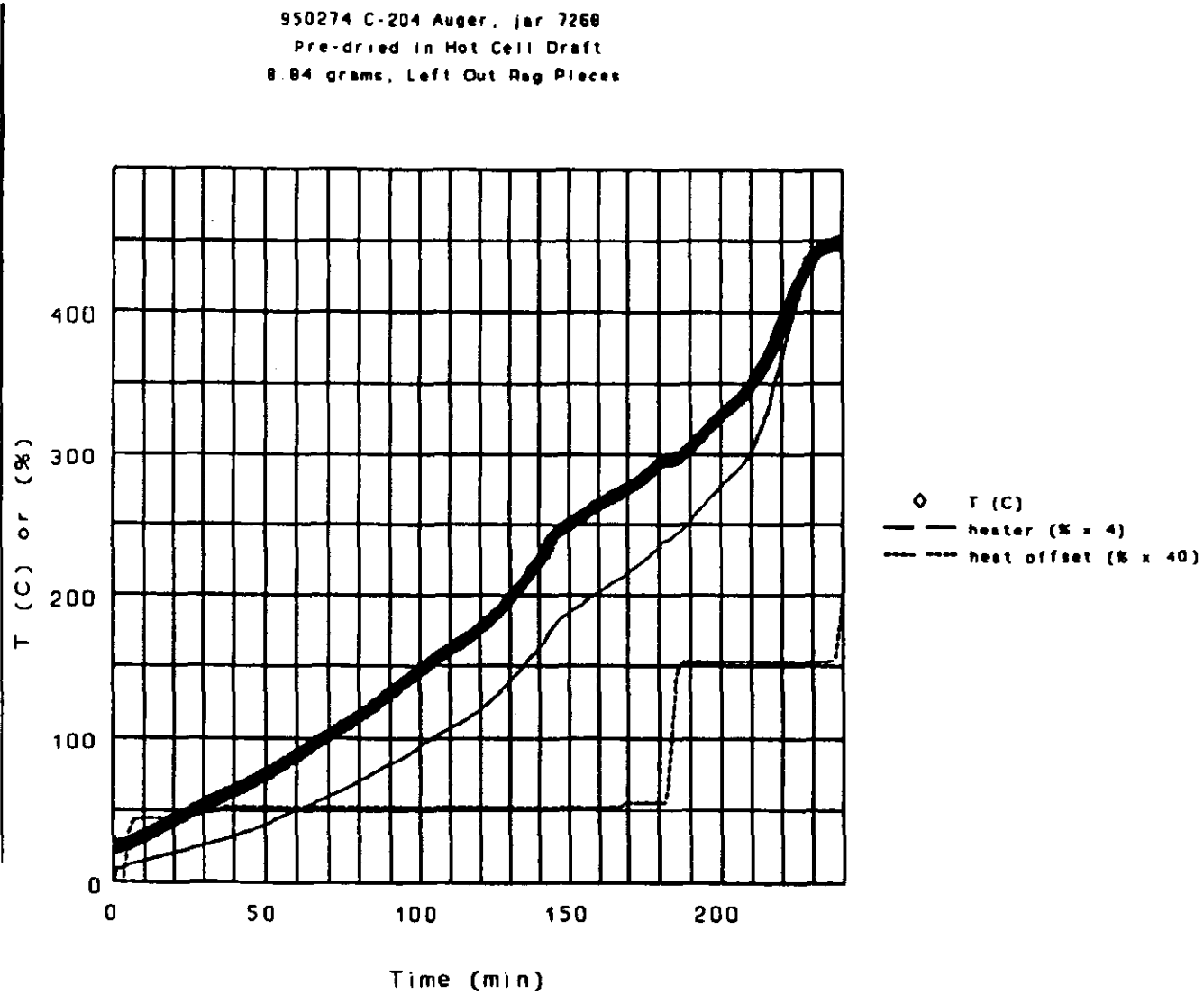


Figure B-5. Temperature Change vs 1/Temperature.

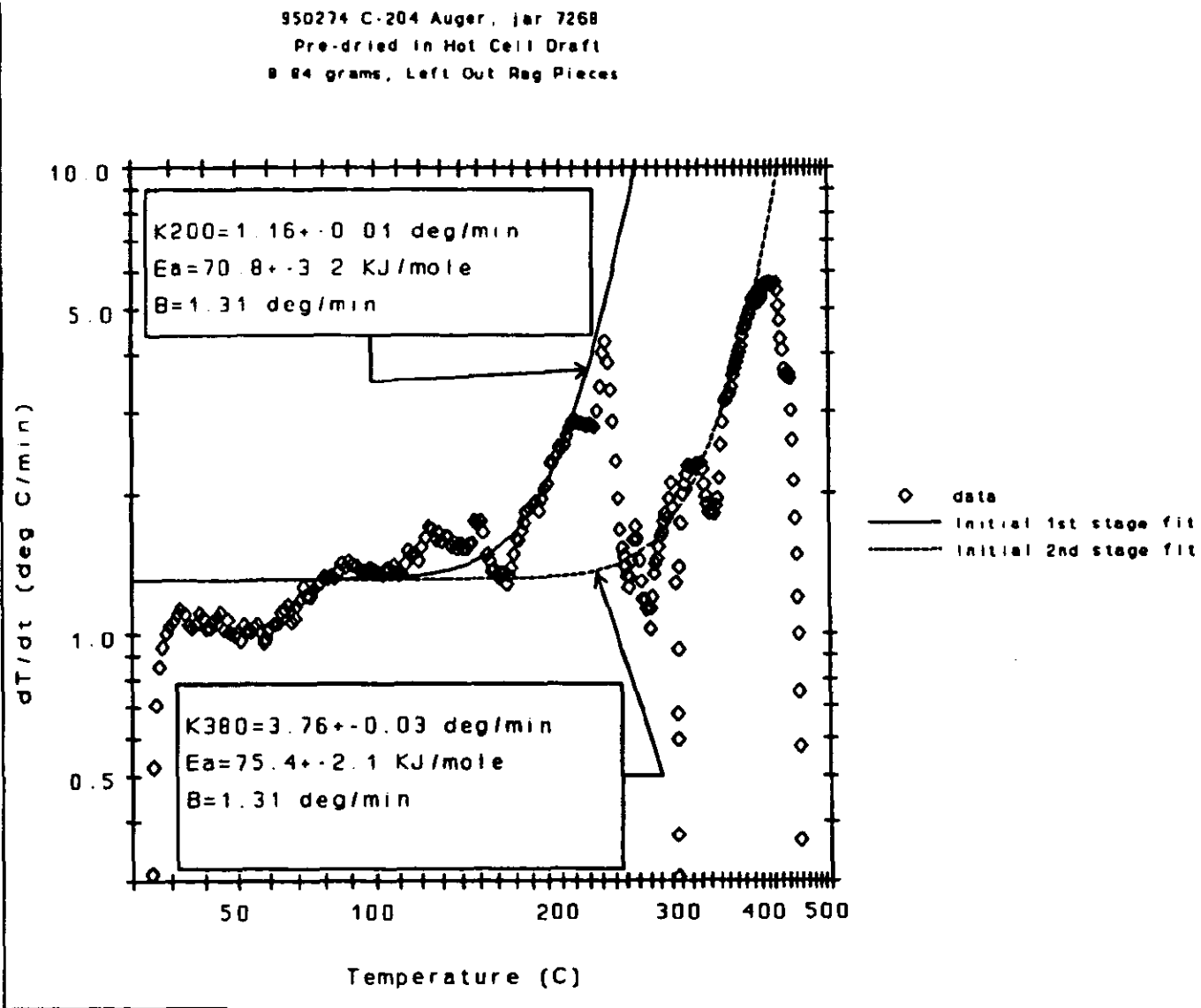


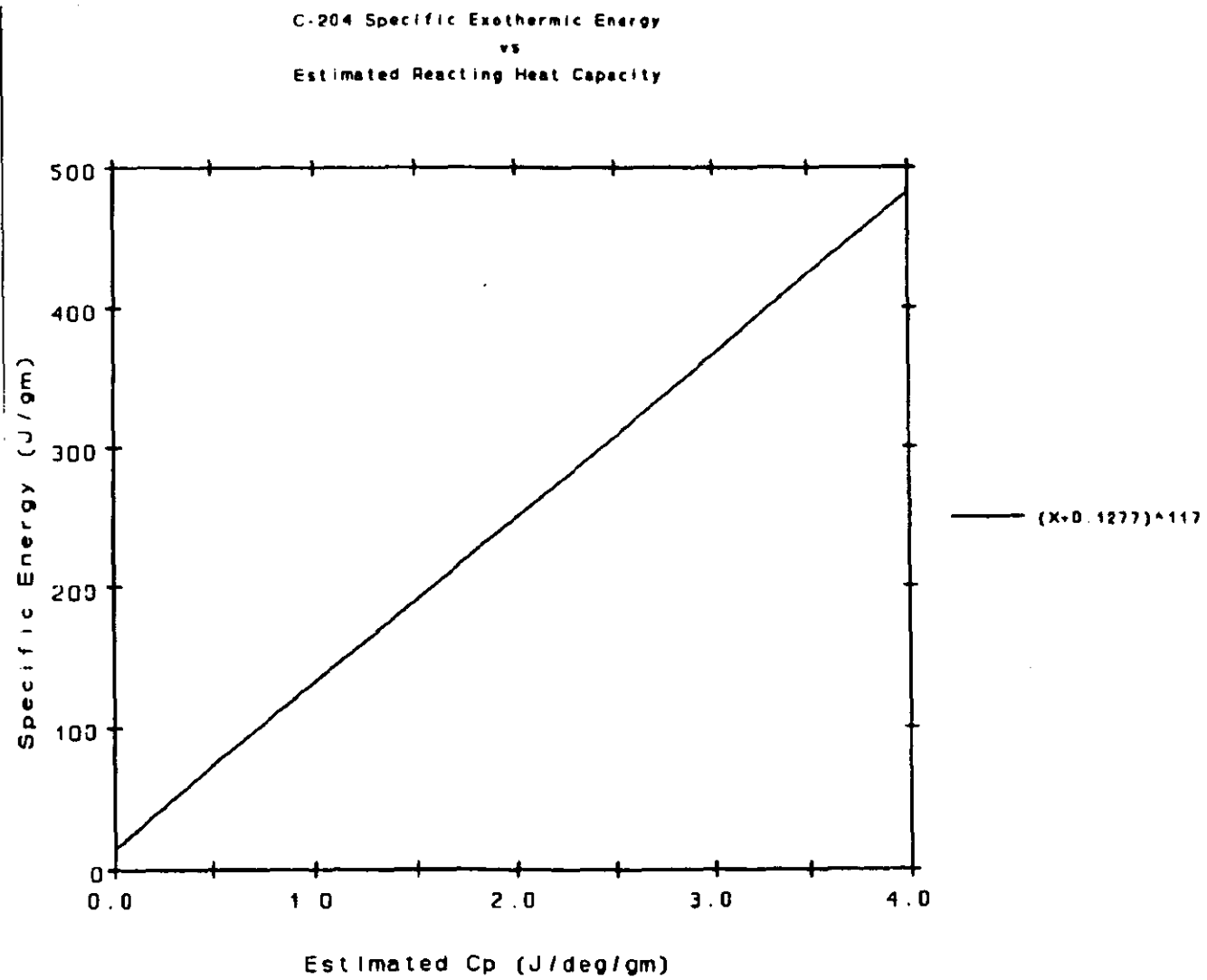
Table B-1. Adiabatic Calorimetry: Tank C-204 Self-Heat Results.

Test ID	T _{start} (°C)	ΔT_{peak}^1 (°C)	Max. dT/dt (uncorr.) (°C/min), at T (°C) ¹	Initial E _i (KJ/mole)	Initial K (C°/min) at T (°C)	Comments
950724	160		$\phi \times 4.3$ °C/min at 234 °C	70.8±3.2	1.16±0.01 at 200°C	1st self-heat event
950724	270		$\phi \times 5.7$ °C/min at 404 °C	75.4±2.1	3.76±0.03 at 380°C	2nd self-heat event
950724	160	$\phi \times 117$ at 450 °C				Overall ΔT for both events

Note:

$$^1\phi = C_{ps}[1 + (0.8368/(C_{ps}w_s))], C_p \text{ in Joule/g/}^\circ\text{C}, w \text{ in grams, } s \text{ denoting sample.}$$

Figure B-6. Adiabatic Calorimetry: Heat Evolution as a Function of Heat Capacity.



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